ANALYTICAL ABSTRACTS

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

2602. **A plan for studying the accuracy and precision of an analytical procedure.** F. J. Linnig, J. Mandel and J. M. Peterson (*Anal. Chem.*, 1954, **26** [7], 1102–1110).—The titration of the fatty-acid in an emulsified synthetic rubber with alcoholic NaOH is taken as an example to indicate the procedure for the examination of the accuracy and precision of an analytical method. Accuracy is investigated by titration of known quantities of fatty acid in the presence of the purified rubber, stabilisers and various quantities of soap, by means of alcoholic 0·1 *N* NaOH that has been standardised against potassium hydrogen phthalate as primary standard. *m*-Cresol purple is used as indicator in all titrations. Results are examined statistically by means of regression analysis. The deviation from linearity of the graph of wt. of material titrated (fatty acid) against ml of reagent (0·1 *N* NaOH) added in presence of stabilisers, and, particularly, various quantities of soaps, is interpreted in terms of pH changes during titration and is consequently ascribed to the behaviour of the indicator. Precision is assessed statistically under similar conditions, with the additional considerations of time, operator and sampling. It is claimed that systematic gross errors of any analysis can be readily segregated by the treatment described whether or not they have any underlying chemical explanation.
D. A. PANTONY

2603. **Separations in one drop.** H. Weisz (*Mikrochim. Acta*, 1954, [1], 140–147).—A simple method of separating ions or groups of ions in a single drop of solution is described. For this purpose two special apparatuses have been devised, one for fixing spots with gaseous reagents, *e.g.*, H_2S, and another, called a ring oven, for washing soluble materials from a spot fleck and concentrating them in a sharply defined circular zone, where they can be detected. The method is exemplified by the separation of Cu, Fe and Ni ions into two groups, *viz.*, Cu and Fe plus Ni, and the separation of Pb, Sb, Fe and Ni ions into three groups, *viz.*, Pb, Sb and Fe plus Ni. The ions are detected within the groups or as individuals by the usual spot tests.
A. J. MEE

2604. **A method of separation [of elements] applied to a single drop. The "ring-oven" method and spot analysis.** H. Weisz (*Mikrochim. Acta*, 1954, [3–4], 376–387).—The ring-oven method previously described (*Anal. Abstr.*, 1954, **1**, 2603) has been used to work out a method of separation of Pb, Bi, Cu, Cd, Sn, Sb, Fe, Co, Ni, Mn, Cr, Zn, Al and Ti. A drop of about 1·5 µl is suitable, if the amounts of cations present are sufficient to permit identification. The cations are separated into three rings and a spot. The test drop is treated with H_2S and washed with 0·1 *N* HCl. The ring contains Fe, Ni, Co, Mn, Zn, Al, Cr and Ti. The spot is then treated first with Br and then with NH_3. Washing with aq. NH_3 gives a ring containing Cu and Cd. The remaining spot is treated with $(NH_4)_2S_x$, which gives a ring containing Sb and Sn, whilst Pb and Bi remain as sulphides in the spot. The cations are then detected by various spot tests.
A. J. MEE

2605. **Physico-chemical analysis of the process of paper chromatography.** H. Erbring and W. Wulf (*Kolloidzschr.*, 1954, **136** [2–3], 158–160).—It is shown that in paper chromatography the adsorption of berberine from 94 per cent. ethanol follows the Freundlich isotherm, but with berberine in 2:4:6-collidine, and with hydrastinine in both these solvents, there is true partition between two liquid phases.
A. B. DENSHAM

2606. **A new technique for quantitative paper chromatography.** I. Mori (*Science*, 1954, **119**, 653–654).—A narrow passage (a quantitative bridge) 2 to 5 mm wide and 30 to 100 mm long (according to the quantity of substance) is made in the centre of the paper strip at the point where the substance should be developed, by cutting out narrow slots with a sharp razor. Heated solid paraffin is absorbed into the paper strip at each end of the bridge so that the developer can pass along the strip only via the bridge. If the developer used dissolves paraffin, the strip must be cut so that only the narrow bridge remains and some other means of reinforcement must be used. When a substance is developed into this bridge, the length of the coloured zone is directly proportional to the concn. of substance and also to the width of the bridge. Optical scanning with a narrow beam of light, whose width is adjusted to that of the bridge, yields quant. results more accurate than those obtained by measuring the irregular form of the coloured zone that is usually developed.
H. F. W. KIRKPATRICK

2607. **Quantitative determination of slightly soluble salts by means of ion exchange.** E. Brochmann-Hanssen (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [5], 307–309).—To a sample of the salt to be analysed is added a weighed quantity of activated ion-exchange resin and a measured vol. of distilled water. After mechanical shaking, the mixture is filtered and the vessel and filter are washed with hot water. The filtrate is titrated with 0·1 *N* NaOH soln. against a suitable indicator. The method has been applied to various "insoluble" chemicals and tablets, including Ca, Pb, Ba and Bi salts, with reproducible and reliable results.
N. M. WALLER

2608. **Spot colorimetry.** H. Weisz (*Mikrochim. Acta*, 1954, [3–4], 460).—The ring-oven method previously described (*Anal. Abstr.*, 1954, **1**, 2603) can be adapted to give a semi-quantitative colorimetric procedure. The ring zone is the same size no matter how many drops are used, and it is therefore possible to develop a number of zones with different numbers of drops and compare the colour intensity with a standard scale.
A. J. MEE

2609. **The colorimetric determination of organic substances with particular reference to biological materials.** W. Krauss and H. Grund (*Z. anal. Chem.*, 1954, **142** [3], 173–189).—The paper is divided into two parts. The first part deals with the theory of absorption spectra of "primary complexes," *i.e.*, those formed by direct bimolecular reaction between the substance to be determined and the reagent. These alone are considered suitable for colorimetric measurements. It must be borne in mind that the position of the absorption maximum depends on the number of resonating π electrons only, and is thus not characteristic of the substance. In biological material in particular other substances with similar π-electron structures may be present. This applies to methods in which complexing agents are used, as well as to methods in which the substance is determined directly. The spectra of polyenes and their dependence on the number of conjugated carbon atoms are chosen as examples and are treated theoretically.

The second part deals with colorimetric estimations not directly involving the substance to be determined but products of a reaction that it has undergone. These are in general considered less suitable for colorimetric estimation as they are less precise and not reproducible; such methods must therefore be examined very critically. A warning is given about methods involving concentrated acids, as these generally fall into the second category The reactions between vitamin D_3 and $SbCl_5$ - acetyl chloride reagent and between ergosterol and the same reagent are given as examples of this type of reaction. P. S. STROSS

2610. **Effect of stray light in prism and grating spectrographs on emulsion calibration curves, and its significance in spectrographic analysis.** R. N. Kniseley and V. A. Fassel (*J. Opt. Soc. Amer.*, 1954, **44** [5], 390–393).—Stray light in Littrow prism spectrographs arises from direct reflection and a scattering of light from the front surface of the collimating-camera lens and from lens flare resulting from internal reflection of light from the lens surfaces. One result is that calibration curves obtained from spectrograms exposed in Littrow prism spectrographs exhibit a smaller slope than curves derived from spectrograms taken by means of grating instruments with a Wadsworth or Eagle mounting. The presence of this stray light will contribute no errors to intensity ratio measurement, provided that the number and spacing of exposures are arranged so that the stray light contribution in the spectrograms used for emulsion calibration is the same as in the spectrograms in which intensity ratios are to be measured. B. S. COOPER

2611. **Effect of inert atmospheres on emission spectra in the interrupted arc.** H. Stone (*J. Opt. Soc. Amer.*, 1954, **44** [5], 411–413).—Atmospheres of helium, helium - oxygen, argon and air have been used in a study of the spectra of various elements excited in the interrupted arc. In general, no significant increase in sensitivity was observed. Be is an exception; the intensity of the sensitive 3130 A doublet is ten times greater in the helium - oxygen atmosphere than in air. B. S. COOPER

2612. **A solution method of spectrographic analysis.** S. Muir and A. D. Ambrose (*J. Iron Steel Inst.*, 1954, **177** [4], 400–405).—The sample (0·5 g) is dissolved in perchloric acid (60 per cent.) if possible, and the solution is evaporated to fuming. After cooling, the perchlorates are dissolved in H_2O and the solution is used to impregnate a prepared graphite electrode. The procedure for choosing a suitable comparison line is described. Details of the procedure for the analysis of low-alloy steels, iron ores, and blast furnace slags are given. The method, although not universal, is of wide application. SiO_2, TiO_2, etc., cannot be dissolved in perchloric acid; fusion with a mixture of Na_2CO_3 and borax, and extraction with dil. HNO_3 may be used to provide the solution for excitation. Results agree satisfactorily with those obtained by the usual chemical methods. A. J. MEE

2613. **Ultra-violet absorption spectra of some inorganic ions in aqueous solutions.** R. P. Buck, S. Singhadeja and L. B. Rogers (*Anal. Chem.*, 1954, **26** [7], 1240–1242).—The u.v. absorption spectra (at 200 to 320 mμ) of aq. solutions of the following ions are described: (*a*) SO_4'', SO_3'', S_2O_3'', S_2O_8'', NO_3', NO_2', ClO_3', ClO_4', BrO_3', IO_3', IO_4', VO_3', WO_4'', MoO_4'', CN', CNO', CNS', PO_4''', H_2PO_2', P_2O_7'''', acetate, citrate, oxalate and tartrate, as K or Na salts (0·1 *M*); (*b*) H_3BO_3 and H_3PO_4; (*c*) Zn^{++}, Co^{++}, Cd^{++}, Hg^{++}, Cu^{++}, Ni^{++}, Mn^{++}, Pb^{++}, Tl^{+}, Ag^{+}, In^{+++}, Ga^{+++}, Fe^{+++}, Th^{++++} and UO_2^{++} (all at $\simeq$ 10 p.p.m.) as perchlorates in 2 *M* H_3PO_4, 0·01 *M* $K_4P_2O_7$, 0·01 *M* sodium citrate, 0·01 *M* sodium tartrate and 0·02 *M* KCN aq. solutions; (*d*) AsO_3''', MoO_4'', WO_4'' and VO_3' in H_3PO_4 and P_2O_7'''' solutions. Only NO_3' (at 198 and 302 mμ), NO_2' (at 210 and 354 mμ), VO_3' (at 266 mμ), IO_4' (at 222 mμ), S_2O_3'' (at 215 mμ), Hg^{++} (at 235 mμ), Ni^{++} (at 270 mμ), Cu^{++} (λ varies with the substrate) and Fe^{+++} (λ varies with the substrate) show absorption maxima. D. A. PANTONY

2614. **Particle size measurements by light scattering.** R. O. Gumprecht (*Dissert Abstr.*, 1954, **14** [4], 646–647).—Light scattering can be used as a means of determining particle size and distribution in atomic sprays. The Beer - Lambert transmission equation must be corrected for the optical geometry of a transmitted-light measuring system. This correction may be predicted from the Mie or, preferably, the classical diffraction theory. T. R. MANLEY

2615. **Photometric titrations in non-aqueous solvents [glacial acetic acid].** C. N. Reilley and B. Schweizer (*Anal. Chem.*, 1954, **26** [7], 1124–1126).—Approx. 0·1 *N* $HClO_4$ in glacial acetic acid is run into the thermostatically controlled spectrophotometer cell containing an 0·05 *N* soln. of quinoline, *o*-chloroaniline, *m*-chloroaniline or sodium acetate in glacial acetic acid. The absorbancy of the soln. is followed during the course of the titration at selected wavelengths, these having been chosen from the comparison of the light-absorption curves of the free organic bases and their salt forms. The two linear limbs of the titration - absorption curves are extrapolated to enable the end-point of the titrations to be found. Comparison is made with potentiometric titrations and agreement is good. Attempted titration of a sodium acetate - *o*-chloraniline mixture with 0·1 *N* $HClO_4$ is not successful. D. A. PANTONY

2616. **Theory of photo-electric complex formation titrations using metal indicators.** A. Ringbom and E. Vänninen (*Anal. Chim. Acta*, 1954, **11** [2], 153–167).—Theoretical conditions are derived for the photometric determination of the exact equivalence point in the titration of metals with ethylenediaminetetra-acetic acid by means of single-colour and two-colour indicators. The principles are

illustrated and confirmed experimentally by the titration of Cu^{++} with murexide as indicator and the titration of Mg^{++} with Eriochrome black T as indicator. W. C. JOHNSON

2617. **A new organic boron complex as an analytical reagent.** R. Neu (*Z. anal. Chem.*, 1954, **142** [5], 335–341).—By the action of HCl on tetraphenyl boron at room temp. a new compound, tetraphenyl diboroxide, is produced. The latter gives intense colours, suitable for colorimetric analysis, with flavonols, aglycones and flavonol heterosides. The colours are stronger than those obtained with boric acid and are stable to water. Morin, quercetin, rutin, quercitrin and hyperin give easily distinguishable colours and fluorescences; these are described. Hypericin changes colour from red to green, but anthocyanins do not react. Nitrogen-containing heterocyclic compounds such as 8-hydroxyquinoline and derivatives are pptd., giving ppt. with characteristic m.p.; 2-hydroxyquinoline does not react. Further investigations into groups of substances that do or do not react are in progress. A simple colorimetric estimation of rutin and quercetin is described. P. S. STROSS

2618. **Structure and behaviour of organic analytical reagents. V. Dimethylglyoxime and its O-monomethyl ether.** R. G. Charles and H. Freiser (*Anal. Chim. Acta*, 1954, **11** [2], 101–110).—The titration procedure described in a previous paper (*J. Amer. Chem. Soc.*, 1952, **74**, 1383) is applied to the metallic complexes of dimethylglyoxime and those of its O-methyl ether. Formation constants are determined for the Cu^{II}, Ni^{II}, Co^{II}, Zn^{II}, Pb^{II}, Cd^{II} and La^{III} complexes of dimethylglyoxime and for the Cu^{II}, Ni^{II}, Mn^{II}, Zn^{II} and Cd^{II} complexes of the methyl ether. The Ni^{II} complex of dimethylglyoxime is not distinguished by exceptional stability, so the selectivity of the reagent for Ni must be due to other factors. The changes in magnitude and order of stability of the complexes that result from the O-methyl substitution are discussed in terms of hydrogen-bonding and steric hindrance. Heat content and entropy changes are calculated for the chelation of dimethylglyoxime with Cu^{++} and Ni^{++}. W. C. JOHNSON

2.—INORGANIC ANALYSIS

2619. **Gasometric method for determination of hydrogen in carbon.** W. G. Guldner and A. L. Beach (*Anal. Chem.*, 1954, **26** [7], 1199–1202).—An apparatus for tensimetric determination of H (as H_2O) in carbon is illustrated and directions are given for its use. Carbon (0·1 g) containing 0·0004 to 3·5 per cent. of H is heated in a platinum bag at 1000° C in a stream of purified oxygen. The water in the issuing gases is condensed and measured manometrically in a calibrated volume at 25° C. Accuracy cannot be assessed, but precision is given as $\simeq \pm 1$ per cent. over the range 0·2 to 3·5 per cent. of H in C, and $\simeq \pm 7$ per cent. over the range 0·0004 to 0·1 per cent. D. A. PANTONY

2620. **Determination by means of deuterium oxide of the humidity of solids not dispersible in water. Application to the study of compounds with macromolecular structure.** R. Viallard and [Mlle.]–. Marchetti (*Chim. Anal.*, 1954, **36** [8], 214–217).—The density of D_2O is determined after contact with a water-containing substance by determination of the equilibrium temp. of a float. The accuracy of this method is discussed. Apparatus is described for investigating the exchange equilibrium of the water and the D_2O between solid and liquid, and solid and vapour phases. Examples are given of results with a synthetic rubber (Perbunan), methyl methacrylate (Plexiglas), and cellulose (in which additional hydrogen exchange occurs, but at a different rate between the D_2O and the mobile hydrogen atoms of the molecule). E. J. H. BIRCH

2621. **The determination of potassium and sodium in coal ash.** R. Belcher, A. J. Nutten and H. Thomas (*Anal. Chim. Acta*, 1954, **11** [2], 120–127).—Methods for the determination of Na and K are reviewed. Coal ash is ignited with $CaCO_3$ and NH_4Cl according to the Lawrence Smith procedure, with a small beaker of water on the lid of the platinum crucible. Sodium is determined in the extract from the ignition cake by pptn. as the triple acetate of Na, Zn and UO_2^{++}; the ppt. is weighed or is titrated with 0·1 *N* NaOH. Potassium is determined by pptn. as $KB(C_6H_5)_4$ and weighing. The results are compared with those obtained by other methods. The flame photometer is much more rapid, but by use of it results are less reproducible. W. C. JOHNSON

2622. **Gravimetric determination of copper as $CuHg(CNS)_4$, and iodimetric determination in presence of [tervalent] Fe, Al or Cr ions.** F. Sierra and C. Abrisqueta (*An. Soc. Esp. Fís. Quím., B*, 1954, **50** [4], 421–426).—The Cu^{II} is pptd. as $CuHg(CNS)_4$ in presence of glycerol and phosphoric acid or H_2SO_4 (according to the other ions present), the ppt. is dissolved in aq. NH_3 and a re-pptn. is effected with $(NH_4)_2Hg(CNS)_4$ in presence of glycerol and acid. The Cu^{II} can be determined either gravimetrically or iodimetrically after treatment of the ppt. with KI.

$$2\ CuHg(CNS)_4 + 10I' \rightarrow 2\ HgI_4'' + Cu_2(CNS)_2 + I_2 + 6\ CNS'.$$

L. A. O'NEILL

2623. **The suitability of different cuprous chloride solutions for the absorption of carbon monoxide in the Orsat apparatus.** H. Mertens (*Gas- u. Wasserfach*, 1954, **95**, 79–83).—Two ammoniacal, two neutral and one (hydrochloric) acid cuprous chloride solutions were tested with respect to absorption velocity and capacity, vapour pressure of absorbent and of substances dissolved in the absorbent, and general behaviour of the absorbent (inclination to crystallise, stability, ease of production). The results indicate that the ammoniacal solutions are the most suitable because of their higher absorption velocity and capacity, and their equality in other respects. B. C. U. R. A.

2624. **Paper-chromatographic separation of metal ions. Semi-quantitative determination of copper, silver and mercury.** A. Weiss and S. Fallab (*Helv. Chim. Acta*, 1954, **37** [4], 1253–1256).—The paper-chromatographic separation of those metals that can be pptd. with H_2S or $(NH_4)_2S$ is discussed, as well as the use of violuric acid and quercetin for identifying colorimetrically μg amounts of the metals so separated. A semi-quant. procedure is proposed. W. J. BAKER

2625. **Analytical differentiation of the carbonates of calcium by physical methods.** T. Pobeguin (*Chim. Anal.*, 1954, **36** [8], 203–210).—The nature of biological deposits of $CaCO_3$ is considered. Optical examination is useful for the qual. examination of precipitates, but is less useful for biological

material. Spectrographic analysis between 2300 and 5800 A gives a qual. estimation of impurities, notably P and Si, present. Debye - Scherrer X-ray diagrams with $Cu_{K\alpha}$ (1·539 A) radiation are sufficiently different for identification of the crystalline forms in inorganic or biological material, but the quant. accuracy is low. The i.r. spectra of calcite, aragonite, amorphous $CaCO_3$, and vaterite are shown, as well as those of mixtures of these with α-$Ca_3(PO_4)_2$. The i.r. spectra of some biological deposits, such as crab claw and cattle bone are recorded, and an estimate of composition is made by comparing the i.r. spectra of similar artificial mixtures. E. J. H. BIRCH

2626. **Separation of barium and strontium on an ion-exchange column.** R. Bovy and G. Duyckaerts (*Anal. Chim. Acta*, 1954, **11** [2], 134–144).—The separation of Ba^{++} and Sr^{++} by means of base exchange resins in the presence of disodium ethylenediaminetetra-acetate (**I**) has been investigated. Salts of radioactive Ba and Sr are used, and the distribution of the ions is determined by measurements of radioactivity. Simple immersion of the resin in a soln. containing Ba, Sr and **I** at various pH values yields partition coefficients that are in accord with values calculated from the stability constants of the Ba and Sr complexes of ethylenediaminetetra-acetic acid (**II**) and the pK values of **II**. Satisfactory separations can be effected in a column by elution with a soln. of **I** under suitable conditions of pH and rate of elution. W. C. JOHNSON

2627. **Micro-analytical separation of barium and strontium by selective enrichment on an aluminium oxide column, and the conductimetric titration of barium.** H. Ballczo and W. Schenk (*Mikrochim. Acta*, 1954, [1], 163–185).—After suitable preliminary treatment, an alumina column can be used to retain Ba only from a neutral solution of the alkaline earths. After removing Ca with HNO_3 by the Rawson - Noll method, the Sr can be determined volumetrically by conversion into borate by the method of Ballczo and Muthenthaller (*Brit. Abstr. C*, 1952, 373). The Ba is dissolved from the column by N HNO_3, the solution is evaporated and treated with a known amount of Ba, warmed, and pptd. by a measured vol. of approx. 0·0004 M K_2CrO_4. After being allowed to settle, the liquid is filtered and the filtrate is back-titrated conductimetrically with 0·001 N $FeSO_4$. The amount of Ba added is treated in the same way. The difference in the two titres gives the Ba content of the sample. The separation is independent of the ratio of concn. of Ba and Sr in the sample. Twenty-five μg of Ba can be determined accurately in the presence of 250 μg of Sr. The method was tested on a "model" water and on two Austrian mineral waters. As the method is sensitive, only a few hundred ml of water are required for a determination. A. J. MEE

2628. **Photometric determination of zinc by means of indo-oxine.** O. Svoboda and W. Prodinger (*Mikrochim. Acta*, 1954, [1], 122–126).—A photometric method for the determination of Zn at concn. of 0·1 to 100 μg of Zn per ml by the indo-oxine reaction is described. It is necessary to adopt slightly different procedures in different concn. ranges. In the range 10 to 100 μg of Zn per ml, deviations from the Beer - Lambert law are considerable, and it is necessary to work at a pH of 3·95 by suitable buffering with Na acetate - acetic acid. For concn. between 1 and 10 μg of Zn per ml, the pH must be 4·50 and for concn. between 0·1 and 1 μg it must be 7·54. When these conditions are met, the accuracy is $\pm$ 1 per cent. A. J. MEE

2629. **Quantitative determination of small quantities of zinc.** A. Lewandowski and W. Kielczewski (*Roczn. Chem.*, 1954, **28** [2], 285–290).—Zinc salt solution is placed with a micro-pipette on a filter-paper strip impregnated with $(UO_2)_2Fe(CN)_6$ or dithizone. The strip is then hung over a hot bath of aq. glycerol (1 + 1) with the lower end touching the surface of the liquid, which diffuses upwards producing a spot, the surface area being proportional to the concn. of Zn in the solution. Estimations of few μg of Zn can be made by this method in under 30 min. with an accuracy of $\pm$ 2 per cent. S. K. LACHOWICZ

2630. **An alkalimetric determination of zinc and lead.** G. Denk and J. Alt (*Z. anal. Chem.*, 1954, **142** [5], 357–360).—Zn and Pb salts can be directly titrated with NaOH by use of cresolphthalein as indicator. The end-point occurs when Zn has been pptd. as $Zn(OH)_2$ and the Pb as $Pb(SCN)_2.Pb(OH)_2$. For zinc, dilute a solution containing 30 to 150 mg of Zn so that the volume after titration is 50 to 70 ml, add 3 g of NaCl, heat to boiling, and titrate with 0·3 N NaOH to a permanent red colour of the cresolphthalein. Sulphate, chloride or nitrate ions interfere only when present at concn. $> M$.

For lead, dilute a soln. containing 0·2 to 0·8 g of Pb so that the volume after titration is $\geq$ 70 ml, add 0·5 to 1·0 g of KCNS and titrate with 0·3 N NaOH to a permanent red colour. Nitrate ions at concn. $>$ 0·3 M cause errors $>$ 1 per cent. The methods described are simple and rapid, but their accuracies are not high. P. S. STROSS

2631. **Line Cd 3261 as internal standard in the spectrochemical determination of zinc on copper electrodes.** J. Ramírez-Muñoz and F. Burriel-Martí (*An. Soc. Esp. Fís. Quím., B*, 1954, **50** [4], 387–398).—Cd and Fe have been considered as internal standards. Sulphate samples and oxide samples diluted with Al_2O_3 and CuO have been examined. The line Cd 3261 appears between two intense Cu lines, and it is necessary to calculate background correction. Results have been best with a cadmium internal standard and CuO as diluting base. L. A. O'NEILL

2632. **The reaction of mercuric ions with *p*-dimethylaminobenzylidenerhodanine and the determination of micro-quantities of cyanide.** O. A. Ohlweiler and J. O. Meditsch (*Anal. Chim. Acta*, 1954, **11** [2], 111–119).—The colorimetric method for the determination of Hg^{++} with *p*-dimethylaminobenzylidenerhodanine (**I**) affords an indirect method for CN$'$, in that the CN$'$ forms a complex with Hg^{++} and only Hg^{++} in excess reacts with **I**. The original procedure of Ohlweiler (*Rev. Bras. Quim.*, 1949, **28**, 23) has been re-investigated. It is shown that precise control of acidity, quantity of reagent, temp. and method of adding the reagent is necessary; the most suitable conditions have been determined. In order to eliminate disturbing effects from the presence of salts and other causes, the soln. (containing up to 3 μg of CN$'$ per ml) is acidified with tartaric acid and the HCN is absorbed in 2·5 ml of a soln. of $Hg(NO_3)_2$ (12·5 μg of Hg^{++} per ml) by the Conway micro-diffusion technique. The diffusion process requires 2 hr. at 27° C or 1 hr. at 38° C. Two ml of the Hg^{++} - CN$'$ soln. are then mixed with 42·5 ml of water and 4 ml of N HNO_3, and 1·5 ml

of a 0·02 per cent. soln. of **I** in ethanol are added from a pipette with shaking. After 20 min. the absorption at 480 mμ is measured and the CN′ is determined by reference to a calibration curve prepared with known quantities of CN′. Temp. must be controlled to within 2° C or a correction must be applied. The standard deviation ranges from ± 0·02 to 0·05 μg of CN′. W. C. JOHNSON

2633. Determination of borate in solutions containing cobalt or chromium. S. Z. Haider (*Analyst*, 1954, **79**, 454–456).—Methods for determination of BO_3''' in presence of Co and Cr are described, the Co being converted to cobalticyanide and the Cr to a soluble chromate. In presence of < 0·06 g of Co, KCN is added until the brown ppt. just re-dissolves, oxidation being completed by addition of H_2O_2 and, after boiling, dil. aq. HNO_3 (1 + 10). The diluted liquid (150 ml) is heated to boiling, the excess of HNO_3 is neutralised and the BO_3''' is determined in the usual manner in presence of glycerol. With excess of Co, the procedure is the same as far as the oxidation with H_2O_2. The intense yellow soln. is acidified with HNO_3 and 0·1 *N* $AgNO_3$ is added in drops until the supernatant soln. is only slightly yellow. The ppt. is coagulated by warming and removed, and the filtrate and washings are used for determination of BO_3''' as before. For mixtures of H_3BO_3 and $CrCl_3$, the soln. is oxidised with Na_2O_2 and the diluted soln. is boiled under reflux. To the cooled soln. dil. HCl and $BaCl_2$ are added and, after further dilution, the soln. is heated to boiling. The cooled soln. is neutralised to methyl red and the BO_3''' is titrated in the usual way. $BaCrO_4$ does not interfere. With mixtures of H_3BO_3 and H_2CrO_4, $BaCl_2$ is added, the soln. is boiled and cooled, the H_2CrO_4 is titrated to the methyl red end-point and the BO_3''' is titrated in the usual way.

A. O. JONES

2634. Study of the colorimetric method of micro-analysis of boron by means of Chromotrope 2B in the presence of acetic anhydride. G. Martin (*Bull. Soc. Chim. Biol.*, 1954, **36** [4–5], 719–729).—Methods of micro-analysis of boron in plants and soils with quinalizarin, carmine and Chromotrope 2B are improved by replacing the H_2SO_4 with a mixture of conc. H_2SO_4, acetic acid, acetic anhydride and H_2O (1:6·2:3·5:0·3 by vol.). Sensitivity is greatest with Chromotrope 2B. A systematic study is made of the effect of various anions and cations on the reaction. Ge^{++++} in 50 times larger quantities gives the same reaction as boron, but its effect can be suppressed by hydroxylamine. Oxidising agents, which interfere with the reaction, are reduced with hydrazine and hydroxylamine. Interference is also caused by phosphates, fluorides and oxides of Fe and Al, which are largely eliminated in the method described, and by Cu, Ni and Co, whose concn. in plants and soils are too low to be important.

C. E. SEARLE

2635. Application of sensitivity diagrams of a reaction to the standardisation of the purity of primary substances. I. Borax. F. Burriel-Martí, J. Ramírez-Muñoz and R. Escobar-Godoy (*An. Soc. Esp. Fís. Quím., B*, 1954, **50** [4], 365–386).—Methods for the detection of Ca (pptn. as oxalate in acetic acid), K (pptn. as cobaltinitrite), Mg (magneson or Titan yellow test), and Cl (pptn. as AgCl) in borax are described. Tests are on a micro scale, ≃ 20 ml of 5 to 10 per cent. borax solution being required for all the tests. Minimum quantities of the impurities that can be detected in the presence of the large excess of borax have been determined and sensitivity diagrams have been constructed. From those diagrams it is possible to obtain a semi-quantitative estimate of the content of impurities from a series of qualitative tests carried out until the limit of detectability is reached.

L. A. O'NEILL

2636. Chromatographic detection of scandium. O. H. Johnson and H. H. Krause (*Anal. Chim. Acta*, 1954, **11** [2], 128–131).—Scandium is separated from other metals by downward diffusion on a 1-in. × 40-cm paper strip. The soln. of metals, as nitrates, is adjusted to pH 2 with HNO_3, and 0·01 ml is applied to the paper, which is dried at room temp. The solvent consists of a mixture of methyl acetate, water and conc. HNO_3 (85:10:5). When the solvent has travelled 30 cm, the paper is dried at room temp., sprayed with a saturated soln. of quinalizarin in 95 per cent. ethanol, suspended in an atmosphere of NH_3 for 5 to 10 min., and air-dried. A number of metals yield colours, and those produced by Zr, Ti, Th and the rare earths resemble the red-violet produced with Sc, but R_F values determined for these metals indicate that Sc is readily distinguishable by separation. The sensitivity limit is 10^{-4} mole of Sc and the method is suitable for the detection of as little as 0·1 per cent. of Sc in ores.

W. C. JOHNSON

2637. The separation of lanthanum and actinium by continuous paper electrophoresis. M. Lederer (*Anal. Chim. Acta*, 1954, **11** [2], 145–148).—The electrolyte (1 per cent. aq. citric acid) diffuses from a trough downwards on a 26·5-cm × 30-cm sheet of filter-paper, and the soln. containing La and Ac (or rare-earth) hydroxides dissolved in 1 per cent. citric acid is applied from a beaker through a tongue cut from the upper edge of the paper. The lower edge of the paper is serrated and each of the two lower corners dips into a vessel containing 1 per cent. citric acid and an electrode. A p.d. of 300 V is applied to the electrodes. Suitable receivers are placed under the tips of the serrations. The electrolyte diffuses at a rate of 100 ml per day. Three ml of a soln. containing 30 mg of rare earths can be separated in 5 days. A complete separation of La and Ac is effected and the tracks of the components can be revealed on the paper by spraying it with alcoholic ammoniacal 8-hydroxyquinoline and examining it in u.v. light.

W. C. JOHNSON

2638. Ion exchange as a separations method. VIII. Relative elution positions of lanthanide and actinide elements with lactic acid eluant at 87° C L. Wish, E. C. Freiling and L. R. Bunney (*J. Amer. Chem. Soc.*, 1954, **76** [13], 3444–3445).—By using Dowex 50 cation-exchange resin and a radioactive tracer technique to determine the relative elution positions, it is shown that the use of lactic acid at 87° C as eluate offers a 4 per cent. increase per column stage compared with 0·25 *M* citric acid in the separation of the tervalent actinide elements. Just as when citric acid is used, Am elutes almost in coincidence with Pm, whilst Cm appears between Sm and Pm. Cf elutes between Dy and Tb with citric acid, but between Tb and Gd with lactic acid.

P. S. STROSS

2639. Fluorescence spectrometric determination of terbium. V. A. Fassel and R. H. Heidel (*Anal. Chem.*, 1954, **26** [7], 1134–1137).—Investigation of the fluoresence of aq. $TbCl_3$ solutions irradiated with a hydrogen arc shows 7 prominent bands at approx. 487, 545, 587, 620, 647, 669 and 618 mμ. Of these

the strongest, at 545 mμ, is chosen for analytical purposes; other rare-earth ions do not interfere at this wavelength. The plot of $Tb^{\cdots}$ concn. against fluorescence power is linear over the range of 0·05 to 2·0 mg of $Tb^{\cdots}$ per 10 ml of soln. Direct measurement of the fluorescence allows determinations precise to 1·8 to 2·5 per cent. $Ce^{\cdots}$, $Cr^{\cdots}$, $Cu^{\cdot\cdot}$, $Fe^{\cdots}$, $UO_2^{\cdot\cdot}$ and NO'_3 interfere in fairly high (≃ 1 per cent.) concn., but their effects are rendered negligible by ignition to 900° C or by treatment of the test soln. on an ion-exchange column. The minimum detectable $Tb^{\cdots}$ concn. is 0·005 per cent., provided that interfering ions are completely absent. D. A. PANTONY

2640. **Self-absorption correction in carbon-14 counting.** E. K. Gora and F. C. Hickey (*Anal. Chem.*, 1954, **26** [7], 1158–1161).—The effect of the geometry of the counting arrangement, the mass absorption coefficient and sample thickness are treated theoretically, and an equation is derived relating these factors to the relative specific activity for weak β-emitters such as ^{14}C. Results are confirmed by conventional measurements on labelled cholesterol digitonide. Standard deviations of the results are reduced to 1·2 to 6·8 per cent., although back- and self-scattering effects are neglected. D. A. PANTONY

2641. **Micro-analysis of germanium.** G. Bartelmus and F. Hecht (*Mikrochim. Acta*, 1954, [1], 148–162).—The use of thioacetamide for the pptn. of GeS_2 has been studied. The pptn. of GeS_2 from boiling H_2SO_4 solution by thioacetamide is not practicable as a macro-method, but satisfactory results are obtained in cold H_2SO_4. If an ammoniacal Ge^{IV} solution is warmed for a short time with thioacetamide, a soluble thiogermanate is formed, and from this solution GeS_2 is pptd. by the addition of a strong acid. Analytically the procedure is not satisfactory, as results are not reproducible. Pptn. from HCl soln. in the cold is a satisfactory macro-method. For the micro-determination, the thioacetamide must be specially purified. Recrystallisation from H_2O is not sufficient; the aq. soln. should be extracted with ether and the extraction repeated. The micro-determination can be carried out from H_2SO_4 soln. with subsequent estimation as oxine - germano-12-molybdate. The quant. distillation of $GeCl_4$ has been studied. It is carried out in a current of CO_2 by the method of Geilmann and Brünger (*Z. anorg. Chem.*, 1931, **196**, 312; *Biochem. Z.*, 1935, **275**, 375). In the macro-determination by means of thioacetamide results were up to 2·4 per cent. too low. Pptn. as oxine - germano-12-molybdate gave results 2 to 7 per cent. too high. Pptn. as pyridine - germano-12-molybdate also gave high results. The last two methods of pptn. were also used on the micro-scale. Good results were obtained by the use of 5:7-dibromo-8-hydroxyquinoline in the gravimetric determination of Ge. This reagent gives a compound with germanium-12-molybdic acid that is not readily soluble. A. J. MEE

2642. **Effect of ashing temperature on the volatility of germanium in low-rank coal samples.** C. L. Waring and W. P. Tucker (*Anal. Chem.*, 1954, **26** [7], 1198–1199).—Three samples of Ge-bearing coal (1 g at < 80 mesh, containing 0·01, 0·6 and 1·1 per cent. of Ge) were heated under various conditions: (*a*) slowly to 200°, 400°, 500°, 800° and 1000° C and then at those temp. for 4 hr., (*b*) rapidly to 800° C and then at that temp. for 4 hr., (*c*) spread in an open dish and rapidly heated to 1000° C and maintained at that temp. for 1 hr., and (*d*) packed tightly in an almost closed crucible at 1000° C for 1 hr. Ge contents measured spectrographically showed no loss under any of these conditions. D. A. PANTONY

2643. **Paper chromatography of inorganic ions. VII. The paper chromatography of germanium.** M. Lederer (*Anal. Chim. Acta*, 1954, **11** [2], 132–133).—Separation of Ge^{IV} from As and other metals by paper chromatography is effected by means of butanol saturated with *N* HCl. The ascending technique is used and the spots are developed: (*a*) by spraying with aq. ammonium molybdate followed by Na stannate in 5 *N* NaOH to give a blue colour; (*b*) by dipping the dried paper in alcoholic ammoniacal 8-hydroxyquinoline, the spot then fluoresces pale green in u.v. light; or (*c*) the paper is exposed to H_2S and then dried; the Ge^{IV} spot is colourless and the As^{III} spot pale yellow; if the paper is now dipped in 0·01 *N* $AgNO_3$ both spots turn black. The following R_F values are recorded: Ge^{IV}, 0·26; As^{V}, 0·84; As^{III}, 0·52. W. C. JOHNSON

2644. **The determination of titanium by high-precision absorptiometry.** W. T. L. Neal (*Analyst*, 1954, **79**, 403–413).—The method described is based upon comparison of a soln. of unknown Ti concn. (coloured by the complex formed with H_2O_2 and H_2SO_4) with a similar soln. of known concn., the transmission ratio of the two soln. giving a measure of the difference in concn. Two methods of comparison of the concn. are available. In one, a calibration graph is constructed with a reference soln. of known concn.; in the other, aliquots of the standard and sample soln., such that both are of approx. the same concn. and so that the concn. of the standard soln. is 15 ± 0·05 mg of Ti per 100 ml, are compared, the difference in concn. of sample and standard soln. being proportional to the difference in transmission ratio. The concn. of the sample soln. is then determined by applying a small correction to the concn. of the standard soln. The former method is convenient for routine work with samples of unknown Ti content and the latter when the approx. Ti content is known. A. O. JONES

2645. **The determination of titanium in uranium - titanium alloys by differential absorptiometry.** G. W. C. Milner and P. J. Phennah (*Analyst*, 1954, **79**, 414–424).—A differential method for determination of Ti in Ti - U alloys is described. A soln. of the alloy in HNO_3 and HF is fumed with H_2SO_4 and diluted, and an aliquot is treated with H_2O_2. A standard soln. of Ti is prepared in the same way as well as a range of standards containing 12 to 19 mg of Ti per 20 ml. A preliminary determination of the Ti content of the alloy soln. is made by comparing its optical density with that of the standard soln., both being measured against a blank containing the acid and H_2O_2. An aliquot of the alloy soln. containing from 12 to 19 mg of Ti is now treated with H_2O_2 and diluted to 100 ml with 2·5 *N* H_2SO_4 (soln. *a*). A similar aliquot is diluted with the acid only (soln. *b*). An aliquot from the range of standards containing as nearly as possible the same amount of Ti as the alloy soln. is treated similarly with H_2O_2 and acid (soln. *c*). The optical density of *b* against the H_2O_2 and H_2SO_4 blank gives the contribution of U to the optical density of the sample. The difference in optical density between *a* and *c* is determined, and, according as *a* or *c* has the higher value, the optical density of

b is deducted from or added to this difference, and the corresponding amount of Ti is found from a calibration graph. Although macro amounts of U can be determined differentially, this was found to be impossible in presence of Ti. A. O. JONES

2646. **Titrimetric determination of zirconium.** J. S. Fritz and M. O. Fulda (*Anal. Chem.*, 1954, **26** [7], 1206–1208).—A soln. (0·003 to 0·005 *M* with respect to $Zr^{\cdots\cdot}$) is adjusted to pH 1·4 with aq. NH_3 and either titrated directly with 0·05 *M* disodium ethylenediaminetetra-acetate (**I**) with Eriochrome cyanine RC as indicator, or 0·05 *M* **I** is added in known excess, and the excess is back-titrated with 0·05 *M* $ZrOCl_2$ soln. with Eriochrome cyanine RC or Alizarol cyanone RC as indicator. The interferences of 47 ions are discussed, and of these $Bi^{\cdots}$, $Hf^{\cdots\cdot}$, $Mo^{\cdots}$, MoO_4'', $Sb^{\cdots}$, $Sn^{\cdot\cdot}$, $Ti^{\cdots}$, $Th^{\cdots\cdot}$, WO_4'', F' and SO_4'' and tartrate interfere in both procedures. Interference from $Fe^{\cdots}$ is avoided by reduction of the original soln. with 10 g of amalgamated Zn at pH < 1·0; after pH adjustment to 1·4, the titration is completed in nitrogen. D. A. PANTONY

2647. **New spot test for tin with morin.** F. Feigl and V. Gentil (*Mikrochim. Acta*, 1954, [1], 90–92).—$Sn(OH)_2$ and $Sn(OH)_4$ give blue adsorption compounds with morin that fluoresce blue-green in the ultra-violet and are unaffected by acetic acid. This reaction can be used as a spot-test for Sn. The sensitivity of the reaction is $pD = 6$. Al, Sb^{III} (but not Sb^{V}) and Zr salts interfere. In the presence of these interfering ions, a solution of the salt in an alkaline sulphide is used. This solution is decomposed with H_2O_2 and the resulting suspension or solution is spotted on to filter-paper. The spot is exposed to NH_3 and then a drop of an acetone solution of morin is added. A. J. MEE

2648. **Detection of tin in minerals and alloys and in silk treated with tin salts.** F. Feigl, V. Gentil and D. Goldstein (*Mikrochim. Acta*, 1954, [1], 93–95).—The adsorption reaction of stannous and stannic hydroxides with morin can be used to detect Sn in minerals, alloys and silk. The Sn is first converted into SnI_4, which is soluble in benzene. If the Sn is present in acid solution, *e.g.*, if the original substance has been dissolved in H_2SO_4, it is only necessary to add an excess of KI and shake with benzene. A spot of the soln. is put on to filter-paper, and after the evaporation of the benzene, the paper is treated with aq. NH_3, aq. Na_2SO_3 soln., morin and acetic acid. A bluish-green fluorescent compound is formed. If Al is present, the spot of the original soln. is treated with NaOH. The aluminate and hypoiodite formed leave the spot by capillary action, whilst the stannate remains adsorbed on the paper. The process is then as described in the preceding abstract (No. 2647). A. J. MEE

2649. **Polarographic study of lead in a potassium thiocyanate supporting electrolyte.** J. O. Hibbits and S. S. Cooper (*Anal. Chem.*, 1954, **26** [7], 1119–1120).—Solubility products (at 25° ± 1° C) of $Pb(CNS)_2$ in various concentrations of KCNS are determined by amperometric titration of the $Pb^{\cdot\cdot}$ with 0·025 *M* $K_2Cr_2O_7$ at −1·0 V. The diffusion current constant also at −1·0 V is determined, and hence the diffusion coefficient is calculated to be $1{\cdot}01 \times 10^{-7}$ cm^2 sec^{-1}. From this the $E_{\frac{1}{4}}$ is deduced as −0·385 V, versus the S.C.E.; this is similar to the value of $E_{\frac{1}{4}}$ for Pb in a non-complexing electrolyte. The reduction of $Pb^{\cdot\cdot}$ at the mercury electrode is a 2-electron change. D. A. PANTONY

2650. **Optimum pH conditions for lead chloride precipitation.** S. Tira (*Ric. Sci.*, 1954, **24** [5], 1072–1074).—The max. pptn. of $PbCl_2$ with HCl at 20° C occurred at pH 0·5 in lead chloride soln. and at pH 0·03 in lead acetate soln. Fricke and Sammet's method (*Z. anal. Chem.*, 1943, **126**, 13) was used to estimate the $PbCl_2$ remaining in soln. M. TADMAN

2651. **New modification of the Unterzaucher method for micro-determination of nitrogen.** W. Manser and A. Egli (*Helv. Chim. Acta*, 1954, **37** [4], 1048–1049).—A modification of the original Unterzaucher apparatus and procedure (*Brit. Abstr. C*, 1951, 482) enables a determination of N in organic compounds (2 to 5 mg) to be effected easily in 15 to 20 min. with an error of ± 0·1 per cent. The arrangement is sketched. W. J. BAKER

2652. **Photometric determination of phosphorus in silicate rocks.** H. Baadsgaard and E. B. Sandell (*Anal. Chim. Acta*, 1954, **11** [2], 183–187).—A method is presented for the determination of P_2O_5 (0·01 to 1·0 per cent.) in rocks, with a max. error of 0·01 per cent. The sample (0·5 g) is brought into soln. by repeated evaporation with aq. HF and HNO_3 and baking at 200° to 250° C; it is finally dissolved in 4 ml of conc. HNO_3 and diluted to 25 ml with water. The soln. is boiled and filtered, and the filtrate is diluted to 50 ml. To 25 ml of the filtrate are added 5 ml of a soln. containing 2·5 g of NH_4VO_3 and 20 ml of conc. HNO_3 per litre, and 5 ml of 5 per cent. aq. ammonium molybdate. The soln. is allowed to stand for 30 min., and its transmittancy is determined at 460 mμ. Results are interpreted by reference to a standard graph prepared with known amounts of P_2O_5. Corrections are determined for Fe and for reagent blank. As, Si and Ge cause negligible errors at the concn. likely to be present. No error arises from the presence of TiO_2 up to 1·6 per cent. or ZrO_2 up to 0·1 per cent. W. C. JOHNSON

2653. **Complexes between molybdophosphoric acid and oxygen-containing organic compounds. Application to the determination of P_2O_5.** R. Vigier (*Bull. Soc. Chim. France*, 1954, **21** [5], 702–706).—Water-sol. mol. complexes between molybdophosphoric acid and sol. O-containing org. compounds (ethanol, dioxan, or acetic acid) show a change in colour from yellow to brown with increasing mol. wt., the intensity of colour depending directly on PO_4''' concn. The light absorption of the complex (PO_4 - MoO_3 - acetic acid) is studied at various concn. of the three components and in presence of HNO_3, to preclude reduction of Mo^{VI}. Max. optical density (0·525) is attained at min. concn. of $4{\cdot}1 \times 10^{-3}$ *M* PO_4''', 102×10^{-3} *M* MoO_3, and 1·25 *M* acetic acid, whilst Beer's law is strictly valid at PO_4''' concn. from 0·81 to $4{\cdot}1 \times 10^{-3}$ *M*. Above these threshold values, increased concn. of MoO_3 or acetic acid do not affect the optical density. A colorimetric method is developed for the determination of P_2O_5 in natural phosphates and citric acid solutions of phosphates. The sample is brought into soln. by treatment with HNO_3 and $HClO_4$ and an aliquot (containing ≃ 30 mg of P_2O_5) of the filtrate is transferred to a calibrated flask (100 ml) with the addition of conc. HNO_3 (10 ml), 2·62 *M* acetic acid (25 ml), and 0·68 *M* sodium molybdate (20 ml). The vol. is made up to 100 ml with distilled water, the soln. is kept for 40 min., and the light absorption of the stabilised complex is then measured at 468 mμ. In presence of alkaline citrate, a preliminary heating with 20 per cent. aq. NaOH is

necessary to remove NH_4^+, whilst 25 ml of 0·98 *M* sodium molybdate is added to the aliquot (which should contain ≃ 9 mg of P_2O_5) on which the optical density is measured at 450 mμ. Interference caused by ClO_4^-, CO_4'', Ca, $Fe^{...}$, Al, SiO_3 and citrate ion are negligible under the specified conditions. W. J. BAKER

2654. **Colorimetric analysis of vanadium in steel.** R. Rosotte and E. Jaudon (*Chim. Anal.*, 1954, **36** [6], 160–161).—One g of the V steel is dissolved in 20 ml of dil. H_2SO_4 (1 + 4) and a little HNO_3. After evaporating to fuming and cooling, the soln. is diluted with 30 ml of water and filtered. $Fe^{...}$ is removed by mercury-cathode electrolysis. Ten ml of conc. aq. NH_3 are added to the soln., which is made up to 100 or 250 ml. An aliquot containing 10 to 40 μg of V is made up to 50 ml, and to this and to standards containing 10, 20, 30 and 40 μg of V are added 1 ml of Fe^{III} soln., 5 ml of 0·05 per cent. *o*-phenanthroline and 10 ml of 2 per cent. ammonium acetate soln. Aq. NH_3 is added, if necessary, to adjust the pH to 5 to 6. Optical densities are measured at 520 mμ. Excess of NH_4^+, SO_4'', $Cr^{...}$, $Mo^{...}$ and $Mn^{..}$ are removed or do not interfere, and reproducibility and accuracy are good. D. A. PANTONY

2655. **Colorimetric determination of niobium by the molybdenum blue method.** G. Norwitz and M. Codell (*Anal. Chem.*, 1954, **26** [7], 1230–1234).—A sample of Ti alloy containing up to 2·5 mg of Nb is dissolved in 5 ml of 48 per cent. HF and 5 ml of HNO_3. Ten ml of 12·6 *N* H_2SO_4 are added and the soln. is evaporated to fuming; after cooling, the residue is taken up in 10 ml of H_2O and 5 ml of dil. HF (1 + 50) and made up to 100 ml with H_2O. A 10-ml aliquot is treated immediately with 10 ml of H_2O, 2 ml of 0·06 per cent. aq. Na_2HPO_4 and 5 ml of 2 per cent. aq. $(NH_4)_6Mo_7O_{24}.4H_2O$. After 15 min., 10 ml of 12·6 *N* H_2SO_4 are added rapidly, and, after a further 30 sec., 3 ml of 0·5 per cent. $SnCl_2$ in dil. HCl. Within 5 min. the colour intensity is measured at 715 mμ, and the Nb concn. is derived from standards that have been treated similarly. Effects of various amounts of H_2SO_4, PO_4''', molybdate, and $Sn^{..}$ and the allowed times of reaction are discussed. W, V, As, Mo, Sb, Ce, Bi, Ba, Pb, Ag, Hg, Se, Fe and Ta all interfere seriously. D. A. PANTONY

2656. **The determination of niobium in stainless steels.** G. W. C. Milner and A. A. Smales (*Analyst*, 1954, **79**, 425–430).—An absorptiometric method for determination of Nb in stainless steels is described. The steel is dissolved in $HClO_4$, the soln. is heated to fuming point, HF is added with a few drops of a ^{95}Nb radioactive tracer soln. and the HF is removed by fuming. Cr is reduced by SO_2, which is then removed by boiling. After addition of Na_2SiO_3 soln. and NH_4Cl, the acidity is adjusted and the Nb is pptd. by tannic acid and cinchonine. The ppt. is ignited and the SiO_2 carrier is removed by treatment with HF and H_2SO_4. The residue is fused with $KHSO_4$, dissolved in tartaric acid soln., and an aliquot is added to a mixture of HCl, acetone, and $SnCl_2$, and treated with KCNS. The optical density of the yellow soln. is determined at 385 mμ and is corrected for the value of a blank soln. prepared from Fe in the same way. The Nb content is then ascertained from a calibration graph. Experimental losses are found by means of a γ-scintillation counter. If the required accuracy does not necessitate addition of the tracer, the initial solution can be simplified by use of HCl and HNO_3, subsequent fuming with $HClO_4$, treatment with SO_2 and proceeding as before. Discrepancies between results obtained by this method and those of a gravimetric one were shown to be due to inclusion of Ta in the gravimetric results. The determination of Ta by radioactivation in the separated mixed oxides is also briefly discussed. A. O. JONES

2657. **A rapid quantitative chemical procedure for analysis for niobium.** F. N. Ward and A. P. Marranzino (*Science*, 1954, **119**, 655).—The thiocyanate colorimetric method is modified to determine Nb in rocks containing 20 to 2000 p.p.m. of the element. The CNS′ reaction is carried out in 4 *M* HCl and 0·5 *M* tartaric acid soln., the CNS′ complex being concentrated by extraction with ether. Ether extraction before reduction and removal of Fe with $SnCl_2$ prevents interference from V. Addition of acetone to the ether extract inhibits polymerisation of the CNS′ and stabilises the colour due to Nb. The colour is measured at 385 mμ and referred to a standard graph. Twenty μg of Nb can be determined in the presence of 1000 μg of Fe, Ti or U, 500 μg of V, or 100 μg of W or Mo, with a precision comparing favourably with spectrographic and X-ray fluorescence techniques. H. F. W. KIRKPATRICK

2658. **Extraction of niobium into di*iso*propyl ketone from hydrochloric acid solutions.** H. G. Hicks and R. S. Gilbert (*Anal. Chem.*, 1954, **26** [7], 1205–1206).—Freshly pptd. $Nb_2O_5.xH_2O$ (≃ 20 mg) containing ^{95}Nb is treated with hot conc. HCl, and completely dissolved by successive cooling, saturating with HCl gas and digestion. The HCl concn. is adjusted to 10 *M*, and the soln. is used as a stock for preparation of solutions of various HCl concn., upon which the efficiency of extraction of Nb with equal volumes of di*iso*propyl ketone is tested. The extraction efficiency is measured by radio-counting, and is found to be almost quant. for 10 to 12 *M* HCl solutions. D. A. PANTONY

2659. **Colorimetric determination of tantalum in titanium alloys.** G. Norwitz, M. Codell and J. J. Mikula (*Anal. Chim. Acta*, 1954, **11** [2], 173–182).—Optimum conditions have been found for the colorimetric determination of Ta by the pyrogallol method, and a procedure is presented for the determination of Ta in Ta - Ti alloys containing 0·05 to 5 per cent. of Ta. The Ta is separated from the Ti by two tannin precipitations with an intervening digestion with tannin. The tannin ppt. is ignited and fused with $KHSO_4$, and the melt is dissolved in ammonium oxalate soln. Pyrogallol is then added and the intensity of the resulting yellow colour is measured. Elements that would be found in the usual Ta - Ti alloys do not interfere, but > 0·5 per cent. of Nb interferes by causing occlusion of Ti by the tannin ppt. and Ti yields a yellow colour with pyrogallol. In these circumstances a sample less than the usual 0·5 g is taken for analysis. Tungsten also interferes if present to the extent of > 1 per cent. W. C. JOHNSON

2660. **Determination of traces of antimony in soils and rocks.** F. N. Ward and H. W. Lakin (*Anal. Chem.*, 1954, **26** [7], 1168–1173).—A sample of rock or soil containing 0·5 to 50 p.p.m. of Sb^{III} (0·2 g at < 80 mesh) is fused with 1·5 g of $NaHSO_4$. When organic matter is destroyed, the melt is cooled and extracted with 6 ml of warm 6 *M* HCl. After solution is complete, 1 ml of 1 per cent. Na_2SO_3 and

3 ml of 6 *M* HCl are added. The soln. is filtered and the residue is washed with hot HCl and water. The cooled filtrate is treated with 3 ml of 3·3 per cent. $Ce(SO_4)_2$ in dil. H_2SO_4 and then with 10 drops of 1 per cent. aq. $NH_2OH.HCl$. After dilution with 45 ml of H_2O, the HCl concn. is adjusted to 1 to 2 *M*, and the soln. is extracted with 5 ml of di*iso*propyl ether. The organic layer is separated and washed with 1 per cent. $NH_2OH.HCl$ in *M* HCl, when it is shaken with 2 ml of 0·02 per cent. rhodamine B in *M* HCl. Its absorption is measured at 545 to 555 mμ, or compared by eye with standards. The $Sb^{\cdots}$ concn. is derived from a calibration curve. Extraction of Sb^{III} from soils is normally > 90 per cent., and the standard deviation of four determinations varies from ± 0·1 to ± 1·1 p.p.m. The extraction - reduction procedure avoids interferences from $Fe^{\cdots}$, $Au^{\cdots}$, $Tl^{\cdot}$, WO_4'', $As^{\cdots}$, $Cu^{\cdot\cdot}$ and $Sn^{\cdots\cdot}$ unless they are in large excess. D. A. PANTONY

2661. **The photometric determination of bismuth with thiourea.** W. Nielsch and G. Böltz (*Z. anal. Chem.*, 1954, **142** [5], 321–329).—The well-known method of determining Bi with thiourea in acid solution is examined. The thiourea concn. in the final soln. must be > 12 per cent., whilst the HNO_3 concn. should lie between 2·2 and 10·0 per cent. The extinction is almost independent of time, but varies with temperature and it is therefore advisable to work in a thermostat. Chloride or bromide ions interfere because they change the position and value of the extinction maximum; this can be compensated for to some extent by selecting a different filter. P. S. STROSS

2662. **Detection of bismuth by dithizone in molten naphthalene.** J. K. Carlton and W. C. Bradbury (*Anal. Chem.*, 1954, **26** [7], 1226–1227).—A 0·025 per cent. soln. of dithizone in molten naphthalene reacts with Bi salts very rapidly (< 10 sec.) to yield a brilliant red colour that is clearly discerned at a concn. of 0·004 μg of Bi. Oxalate and phosphate prevent the normal response of Bi to the reagent, and Cd (sensitivity 0·02 μg), Hg (0·01 μg), Sn (0·004 μg), Ag (0·02 μg) and Zn interfere by masking the colour of the Bi - dithizone reaction. Cyanide ion and chloride ion mask the interference of Hg and Ag, respectively, without reduction in sensitivity. D. BAILEY

2663. **Analysis of gases in steels: oxygen.** C. Artero Soteras (*Inf. Quim. Anal.*, 1954, **8** [3], 90–99, 101).—Methods of analysis are grouped according to whether they give (*a*) combined oxygen, (*b*) dissolved oxygen, or (*c*) total oxygen. This report deals with (*a*) and relies on the separation of inclusions of the various oxides (SiO_2, Al_2O_3, MnO and FeO) by chemical attack, either with acids (HNO_3, HCl or a mixture of H_3PO_4 and HNO_3) or with halogens. The former method was considered limited in its application and the main work describes the use of chlorine and iodine in the removal of Fe, to leave the inclusions behind. The use of chlorine had been studied by Colbeck, Craven and Murray (*J. Iron & Steel Inst.*, 1936, **2**, 251) and iodine by Cunningham and Price (*Ind. Eng. Chem., Anal. Ed.*, 1933, **5**, 27). In this method the solution of the iron takes place by heating in a current of dried pure chlorine. The ferric chloride formed is sublimed at the temperature of the furnace and is carried away by the current of gas. At the same time certain substances such as sulphur and carbon are also removed leaving behind unaltered oxides. With iodine, either aqueous or alcoholic methods are used; in the former, a solution of ferrous iodide is the solvent for the iodine. The steel is dissolved in the cold and the residue is washed with ammonium citrate, calcined, weighed and subjected to analysis. If the steel contains more than 0·25 per cent. of Si, it is necessary to wash it with a hot 10 per cent. soln. of sodium carbonate to eliminate the Si. Details are also given of the alcoholic iodine method (Rooney, *Iron & Steel Inst. Special Report No.* 25, 1939, 141) and of the precautions to be taken with steels of high carbon and phosphorus content, and finally, a scheme for separating the various constituents of the residues is given. H. PRITCHARD

2664. **Colorimetric determination of alkali sulphides.** V. Vašák and V. Machálek (*Chem. Listy*, 1953, **47** [6], 850–852).—Sodium diethyldithiocarbamate precipitates Cd as the colourless Cd diethyldithiocarbamate. Addition of $Cu^{\cdot\cdot}$ to the $CHCl_3$ extract of the Cd complex quantitatively expels Cd with the formation of the coloured Cu diethyldithiocarbamate, which can be determined colorimetrically. Decrease in absorption values after addition of the sulphide-containing sample to the soln. of Cd salt is equivalent to the decrease of Cd in soln. and hence indirectly gives a measure of S″. *Procedure*—In a separating funnel, mix a definite vol. of a soln. containing 0·2282 g of $3CdSO_4.8H_2O$ in 1 litre of H_2O (soln. **I**) with 10 per cent. aq. Na K tartrate (10 ml), 10 per cent. aq. NaOH (10 ml) and 1 per cent. aq. Na diethyldithiocarbamate (1 ml). Extract with $CHCl_3$ (10 ml) during 1 to 2 min., filter the organic layer into a second funnel, shake with 10 per cent. aq. $CuSO_4$, filter into a cell and measure the extinction at 535 mμ (value *A*). To an equal vol. of **I** add the sample containing S″ and follow the above procedure regardless of separated CdS (value *B*). Value for S″ is obtained by multiplying (*A* − *B*) by 0·5043. G. GLASER

2665. **The indirect titrimetric determination of the sulphate ion using ethylenediaminetetra-acetic acid.** R. Belcher, D. Gibbons and T. S. West (*Chem. & Ind.*, 1954, [28], 850–851).—Possible applications are reported of a titrimetric method described previously (*Anal. Abstr.*, 1954, **1**, 448) of evaluating $BaSO_4$ precipitates. More rapid methods of pptn. are described and a further procedure is given for indirect determination of the sulphate ion by means of a modification of the method devised by Munger, Nippler and Ingols (*Brit. Abstr. C*, 1951, 127), which involves titration of ethylenediaminetetra-acetic acid with a Mg solution. A standing time of 1 to 2 hr. is adequate for 1 to 2-mg amounts of S, whilst 10 to 15 min. suffices for 30 to 50-mg amounts. With this reduced standing time, the original method can be used successfully to determine S in steel or organic compounds. L. F. TAYLOR

2666. **Chloramine-B as a volumetric reagent. Determination of sulphur compounds.** Apar Singh (*J. Indian Chem. Soc.*, 1954, **31** [4], 327–328).—The use of chloramine-B in the determination of S″, CNS′, SO_3'', HSO_3', S_2O_3'' and S_4O_6'' is reported. *Procedure*—Place a known volume of the soln. in a 500-ml flask fitted with a ground-glass stopper carrying a dropping funnel. Add an excess of 0·1 *N* chloramine-B and 2 to 3 g of KBr. Evacuate the flask and add 20 to 25 ml of HCl for every 100 ml of soln., without admitting air. When acid, set aside for 20 to 30 min. Wash the funnel, again excluding air, add 2 to 3 g of KI and titrate with 0·1 *N*

$Na_2S_2O_3$. For SO_2, HSO_3' and SO_3'', the HCl is added first to the chloramine-B and KBr, and then the HSO_3' soln. is added until the Br is nearly decolorised; the method is completed as before. Results obtained and relevant factors are given.
G. B. THACKRAY

2667. **Determination of hydrogen sulphide in mine damp.** W. Brösse (*Glückauf*, 1953, **89**, 1059–1061).—Hydrogen sulphide in mine gases is determined by its reaction with Cd acetate soln. A portable apparatus for removing completely small amounts of H_2S from large volumes of air is described and illustrated. N. E.

2668. **Sulphur in coal. II. Determination of total sulphur in coals rich in this element.** V. Gomez Aranda and J. Auria Arbunies (*Combustibles*, 1954, **14**, 72–79).—Results by the Eschka method compare favourably with those obtained by a bomb calorimetric or by a Carius method. From 4 to 5 g of reagent should be used for a sample wt. of 0·5 g, when the S content is > 5 per cent. There is no evidence for loss of S on rapidly heating the sample with reagent (1 hr. at 800° C). Results are also satisfactory for cokes and semi-cokes.
L. A. O'NEILL

2669. **Photometric determination of uranium with quercetin.** J. Komenda (*Chem. Listy*, 1953, **47** [4], 531–533).—Quercetin reacts with $UO_2^{\cdot\cdot}$ with the formation of a rusty-brown stable coloration. Maximum sensitivity is achieved at pH 7. Ions reacting with quercetin, especially Fe, must be absent. The method determines 0·005 to 0·8 mg of U with an accuracy of ± 2 per cent. To 0·1 *N* ammonium acetate (10 ml, pH 7·0), add 96 per cent. ethanol (10 ml), followed by neutral aq. soln. of sample containing 5 to 300 μg of U (up to 5 ml) and 0·1 per cent. ethanolic soln. of quercetin (0·5 ml). Make up to 27 ml with water and measure the extinction after 10 min. For samples containing 0·1 to 0·8 mg of U, increase the volume of quercetin soln. to 2 ml and make up to 25 ml. G. GLASER

2670. **Paper-chromatographic determination of uranium [in rocks].** H. Seiler, M. Schuster and H. Erlenmeyer (*Helv. Chim. Acta*, 1954, **37** [4], 1252–1253).—The qual. and quant. method described depends on addition of Na_2CO_3 to the dil. HNO_3 soln. of the sample (after fusion), whereby migration of $Fe^{\cdot\cdot\cdot}$ and $Cu^{\cdot\cdot}$ is inhibited and a sharp separation of the U spot (revealed by treatment with 5 per cent. aq. potassium ferrocyanide) is obtained. The U concn. is obtained by comparison of the area of the spot with that for standard U solutions. W. J. BAKER

2671. **Elution chromatography with thick filter-paper [for the separation of uranium].** W. J. Frierson, P. F. Thomason and H. P. Raaen (*Anal. Chem.*, 1954, **26** [7], 1210–1211).—An apparatus is described and illustrated for the separation of metal ions by elution chromatography with thick filter-paper. The procedure for pre-washing the paper with ether - HNO_3 solvent to remove metal ions and soluble organic matter is described. The method, which is used for a single sample or, by suitably cutting the paper, for the simultaneous chromatography of two samples, is evaluated for the separation of U^{VI} from two synthetic test solutions containing U^{VI}, Cu^{II} and Al. Recovery of U, determined either polarographically or volumetrically, is good. The eluate contains about 0·1 per cent. of Ru, Zr and Nb, if these are present in the original solution. D. BAILEY

2672. **Heterometric micro-determination of [sexavalent] uranium by precipitation as phosphate.** M. Bobtelsky and M. Halpern (*Anal. Chim. Acta*, 1954, **11** [2], 188–191).—A soln. containing $UO_2^{\cdot\cdot}$ (1 to 8 mg) in 0·1 *M* acetic acid (10 ml) containing 4 *M* NH_4Cl (0·5 ml) is titrated with 0·1 *M* to 0·004 *M* Na_2HPO_4 to the initial point of max. turbidity (*cf. Anal. Abstr.*, 1954, **1**, 901, and 1770). The use of pyridine, quinoline or quinine in place of NH_4Cl renders the method more sensitive.
W. C. JOHNSON

2673. **Use of complexones in chemical analysis. XXXVIII. Determination of uranium by the titration of ammonia with hypobromite.** I. Sekerka and J. Vorlíček (*Chem. Listy*, 1953, **47** [4], 512–515).—Addition of NH_3 to a soln. of $UO_2^{\cdot\cdot}$ precipitates U as ammonium uranate (constitution uncertain but NH_4 to U ratio is found to be 1 to 1), in which NH_3 and hence U is determined potentiometrically or polarographically with nascent hypobromite ion. The presence of the complexone during pptn. suppresses interference by a number of accompanying ions. *Procedure*—To the sample, add a 5 per cent. soln. of disodium ethylenediaminetetra-acetate and then aq. NH_3 in slight excess. Filter off the ppt. after 1 hr., remove all $NH_4^{\cdot}$ by washing with ethanol, dissolve ppt. in saturated $NaHCO_3$ soln., add KBr (0·5 g) and titrate with 0·05 *N* $Ca(OCl)_2$ (1 ml ≡ 7·937 mg of U). Characteristic titration curves are given. Be and Ti do not interfere; V gives high values. The method enables U to be determined in the presence of PO_4''', but in that case Be must be absent.
G. GLASER

2674. **Separation of iodide, bromide and chloride from one another and their subsequent determination.** T. J. Murphy, W. S. Clabaugh and R. Gilchrist (*J. Res. Nat. Bur. Stand.*, 1954, **53** [1], 13–18).—To separate iodide, bromide and chloride, the iodide is first oxidised to free I by H_2O_2 in weakly acid solution (pH 1, H_3PO_4). The I is then removed by distillation. Under these conditions negligible amounts of Br and Cl are lost, but if small amounts of I are to be determined in the presence of a large amount of Br, the distillate must be again treated with H_2O_2 and H_3PO_4 and redistilled. Next, bromide is oxidised to Br without affecting the Cl by treatment with dil. HNO_3 [(1 + 3) to (1 + 6)]. Br is distilled off, and the Cl remains in the residue. The distillation apparatus is described. The determination of the separated halides is on the usual lines. If traces only are present a turbidimetric method is used; otherwise potentiometric titration with $AgNO_3$ is used.
A. J. MEE

2675. **New procedure for the titration of alkali halides with silver nitrate in aqueous medium.** G. Laustriat, Y. Volmar and M. Hasselmann (*Ann. Pharm. Franç.*, 1954, **12** [3], 161–171).—The equivalence point in a titration of alkali halides with $AgNO_3$ is shown by the increase in the scattering of light by the flocculating soln. at the end-point. A convergent beam of light passes through the titration beaker (5 cm in diameter) and is received on a screen. When chlorides are titrated, the colour of the spot changes gradually through yellow and orange to a dark red at the end-point; extinction is complete on stirring for a few seconds. With bromides, the spot of light becomes suddenly red at the end-point. With iodides, the spot turns from yellow to red and back to yellow at the end-point, as the micelles

crystallise. Results are best on titrating 0·001 *N* halide with 0·04 *N* $AgNO_3$, higher concn. of halide giving a positive and lower ones a negative error. The reverse titration with 0·001 *N* $AgNO_3$ and 0·04 *N* halide produces similar results, but the chloride and iodide solutions are less stable. The effect of altering pH (below 7, at which Ag_2O is pptd.) and of adding KNO_3 are also investigated.

E. J. H. BIRCH

2676. **Rapid spectrophotometric determination of fluoride with zirconium - Eriochrome cyanine R lake.** S. Megregian (*Anal. Chem.*, 1954, **26** [7], 1161–1166).—The sample containing $\not>$ 0·07 mg of F′ per 50 ml is treated with 5 ml of 0·18 per cent. aq. Eriochrome cyanine R and then with 5 ml of ZrO·· reagent [0·0265 per cent. $ZrO(NO_3)_2$ or 0·022 per cent. $ZrOCl_2$ in dil. HCl (7 + 3)] at standard temp. (22° to 28°C, constant to 2° C). The absorption is measured at 525 to 530 mμ with reference to a blank. The F′ concn. is derived from a calibration curve of F′ concn. plotted against absorption; the graph is prepared at the temp. at which the determination is done. Ions that are commonly present do not interfere at the acid concn. given, and procedures allowing determinations to be carried out in the presence of $Al^{···}$ and SO_4'' are described. Precision is given as ± 0·0163 mg of F′ per litre.

D. A. PANTONY

2677. **Mercurimetric determination of chlorides.** E. G. Hill (*Chem. & Ind.*, 1954, [28], 852–853).—The method proposed by Ungar (*Anal. Abstr.*, 1954, **1**, 1840) for determining chlorides by titration with mercuric nitrate has been investigated, the pH of the solution being varied with different amounts of the buffer solution recommended by Thiel, Schulz and Koch (*Z. Elektrochem.*, 1934, **40**, 150). The colour intensity varied widely over the pH range 4·0 to 6·0, and at pH values above 7·5 the solution became more intensely red as alkalinity increased.

L. F. TAYLOR

2678. **Mercurimetric determination of chlorides.** J. Ungar (*Chem. & Ind.*, 1954, [27], 787).—The mercurimetric determination of chlorides (Ungar, *Anal. Abstr.*, 1954, **1**, 1840) is modified for use in water analysis. When $Hg(NO_3)_2$ is prepared in *N* HNO_3, instead of in water containing 20 ml of 2 *N* HNO_3, accurate results are obtained with a 100-ml water sample and 5 ml of diphenylcarbazone indicator (0·1 g per 100 ml of ethanol). The chloride content of acid solutions, *e.g.*, dil. HCl, can be determined by this method. The lower pH limit of the method can be varied by lowering the pH value of the $Hg(NO_3)_2$ titrating solution.

I. JONES

2679. **Colorimetric determination of iron with meconic acid.** G. Mannelli and R. Biffoli (*Anal. Chim. Acta*, 1954, **11** [2], 168–172).—The red colour resulting from the reaction of meconic acid with $Fe^{···}$ is that of a complex containing 1 mol. of meconic acid to each atom of Fe. The absorption max. of the complex is 475 mμ when the pH of the soln. is within the range 0·8 to 3·6, and at pH 1 the optical density conforms to Beer's law for 0 to 30 μg per ml concn. of Fe^{III}. The limit of etection of Fe^{III} is 0·2 p.p.m. in a 50-cm cylinder; on a spot plate, 0·5 μg of Fe^{III} can be detected in a concn. of 2·5 p.p.m.

W. C. JOHNSON

2680. **Titrimetry of iron after preliminary reduction with stannous chloride.** P. Wehber (*Angew. Chem.*, 1954, **66** [10], 271–273).—The inconveniences inherent in the usual method of determining Fe^{III} by reduction with Sn^{II} and oxidation of the excess of Sn^{II} with $HgCl_2$ are avoided by titrating the excess of Sn^{II} with standard potassium dichromate and use of cacotheline (which can be added before the reduction) as redox indicator; the end-point is sharp if sufficient NH_4Cl is present. Ammonium fluoride is then added until the violet colour of cacotheline reappears, and the Fe^{II} is titrated with the dichromate by using diphenylamine, sulphonic acid as indicator. The coefficient of variation is 0·3 per cent. Cu^{II}, V^{V}, Mo^{VI}, U^{VI} and the complexing anions F′, PO_4''', NO_3' and acetate interfere.

R. C. MURRAY

2681. **A new method of estimating cobalt with nitrilotriacetic acid.** W. Nielsch and G. Böltz (*Z. anal. Chem.*, 1954, **142** [5], 329–334).—The red complex formed from cobalt and nitrilotriacetic acid (**I**) is suitable for colorimetric estimations. The absorption maximum is at 510 mμ and is dependent on pH, being constant from pH 4·88 to pH 5·69. Tartrate and acetate both increase formation of colour and carefully controlled amounts should be added. Excess of **I** does not interfere. The Beer - Lambert law is obeyed for Co concn. from 0·25 to 3·75 mg per ml. The method is suitable for the determination of Co in alloys rich in Co.

P. S. STROSS

2682. **Substitutes for the Zimmermann - Reinhardt reagent in the determination of iron in haematite and magnetite ores.** K. M. Somasundaram and C. V. Suryanarayana (*Proc. Indian Acad. Sci., A*, 1954, **39**, 41–44).—Na acetate and borax were effective as substitutes for the Zimmermann - Reinhardt reagent in pure ferrous iron solutions, but ineffective with extracts obtained from haematite and magnetite. A number of inorganic salts were investigated and were also found to be unsuitable. Manganous acetate and K_2SO_4 were satisfactory. See also *Anal. Abstr.*, 1954, **1**, 1843.

G. C. JONES

2683. **The mercurous salts as new reducing agents for volumetric estimations in alkaline medium. I. Estimation of the ferricyanide ion.** F. Burriel-Martí, F. Lucena-Conde and S. Arribas-Jimeno (*An. Soc. Esp. Fis. Quim., B*, 1954, **50** [3], 289–302).—Stable complexes with mercurous salts are formed by I′ in alkaline medium, and ferricyanide can be accurately estimated by titration of solutions containing ferricyanide, NaOH and KI with a solution of mercurous nitrate or perchlorate, the optimum conditions being pH $\not<$ 14, KI solution of molarity at least 0·2 added immediately before the titration, temp. $\not>$ 30° C, and mercurous salt soln. at an acidity great enough to prevent pptn. of basic salts but at > pH 1. Barium diphenylaminesulphonate is a suitable indicator.

M. TADMAN

2684. **Use of complexones in chemical analysis. XXXIX. Colorimetric determination of cobalt in nickel and nickel salts.** R. Přibil, M. Kobrová and J. Jeník (*Chem. Listy*, 1953, **47** [6], 842–845).—The previously described method for the colorimetric determination of Co in the form of an ethyl acetate extract of Co diethyldithiocarbamate (Přibil *et al.*, *Chem. Listy*, 1952, **46**, 603) has been adapted for the determination of Co in Ni and Ni salts. In the procedure, $Ca^{··}$ liberates Co from the complex of Ni and Co exclusively; the Co is then pptd. with Na diethyldithiocarbamate, extracted with ethyl acetate and determined colorimetrically at 425 mμ. *Procedure*—Dissolve the Ni salt in a small amount of water, add an excess of 5 per cent. soln. of complexone III, make alkaline

with NH_3 and add 0·1 *M* $Ca(NO_3)_2$ followed by freshly prepared 2 per cent. Na diethyldithiocarbamate (2 ml). Bring the soln. to the boil, extract the ppt. after cooling with 2 portions (15 and 10 ml) of ethyl acetate, wash the organic layer with a little water containing 2 per cent. $HgCl_2$ (1 ml) in order to remove traces of Ni diethyldithiocarbamate, and make up to 25 ml with ethanol. G. GLASER

2685. **Intensity measurements in the arc spectrum of nickel.** R. L. Heid and G. H. Dieke (*J. Opt. Soc. Amer.*, 1954, **44** [5], 402–410).—The relative intensities of 478 lines in the arc spectrum of nickel have been determined photo-electrically, the wavelength range being 3359 A to 5893 A. Calibration of the apparatus was effected by using a standardised tungsten ribbon filament lamp whose spectral characteristics were known accurately.
B. S. COOPER

2686. **Use of nickel ferrocyanide ammine in qualitative analysis.** A. S. Kozlov (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1954, **94** [4], 705–706).—A precipitate of green nickel ferrocyanide dissolves in conc. ammonia and the ammine crystallises from the solution after 1 to 2 min. as pale-violet well-formed slender prisms that attain a length of about 100 μ when kept some time in the mother liquor. Their use as a means of characterising nickel is aided by the fact that they decompose with loss of ammonia in the air, with re-formation of the original green ferrocyanide. A suggested procedure is to evaporate a drop of test solution almost to dryness on an object glass and then treat it with a solution of $K_4Fe(CN)_6$ in conc. aq. NH_3 (about 20 per cent.), or to add conc. aq. NH_3 followed by a crystal of solid $K_4Fe(CN)_6$. The alkali and alkaline-earth metals and copper do not interfere, but Cd^{++}, Ag^{+}, Zn^{++} and Mn^{++} do. Co does not interfere unless the Co to Ni ratio is > 2. R. C. MURRAY

2687. **The separation of the platinum metals by means of strongly basic anion-exchange resins.** E. Blasius and U. Wachtel (*Z. anal. Chem.*, 1954, **142** [5], 341–356).—Solutions of salts of the platinum metals are passed down a column of an ion-exchange resin such as Permutit ES in its basic form. The liberated hydroxyl ions react with the metal or metal complex ions. Ru and Rh are precipitated on the column as hydroxides; Pd, Ir and Pt are retained as complex chlorides. Pd can be eluted by hydroxyl ions, Ir after reduction to Ir^{III}, whilst Pt changes partly to hydroxide but is retained on the column. The complex chlorides are held more firmly on the chloride form of the resin and can then only be eluted by large volumes of strong mineral acids.

The separation of the following pairs of metals is described in detail. *Pd from Pt*—$(PdCl_4)''$ is quant. eluted with NaOH whilst $(PtCl_6)''$, which changes to a partial hydroxide, remains on the column and is later stripped with 2·5 *N* HNO_3. After separation, the Pd contains $< 0{\cdot}5$ per cent. of Pt, whilst the Pt contains $< 0{\cdot}01$ per cent. of Pd. *Ir from Pt*—Ir is first reduced to the tervalent form by Na oxalate and the mixture is applied to the column. Ir is quant. eluted with NaOH, the Pt being stripped as before with 2·5 *N* HNO_3. The Ir is contaminated with $< 0{\cdot}1$ per cent. of Pt. *Rh from Pt*—Rh is quant. eluted with NaOH and the Pt then stripped as before. Only traces of Pt remain in the Rh fraction. *Rh from Ir*—Complete separation is not possible.

A method of removing on an ion-exchange column of Permutit RS traces of Fe, Cu and Ni from any of the salts of the Pt metals by absorption of the former is also described. P. S. STROSS

2688. **The microchemical electrolytic analysis of alloys in hydrochloric acid solutions. I. Brasses and bronzes.** A. J. Lindsey and E. A. Tucker (*Anal. Chim. Acta*, 1954, **11** [2], 149–152).—The methods of Lindsey and Sand (*Analyst*, 1935, **60**, 739) for electrolytic separation by graded potential have been applied to the micro-analysis of brasses and bronzes. *Procedure*—Dissolve the sample (5 to 20 mg) in aq. HCl with the addition of $KClO_3$ and evaporate almost to dryness. Add 1 ml of conc. HCl and 5 drops of 50 per cent. aq. hydrazine hydrate and sufficient water to cover the electrodes. Heat the soln. to $\simeq$ 80° C, stir with H or N, electrolyse at 0·8 V until the current falls to one-tenth of its original value and then at 0·85 V until the current is $\simeq$ 3 mA. Wash and dry the cathode by the usual method and determine the wt. of the deposited Cu. Deposit the Sn and Pb on the Cu-coated cathode at 40° C and 1·3 to 1·4 V (15 min.). Cool the soln., add 1 drop of bromophenol blue soln. and aq. hydrazine hydrate to produce a blue colour. Wash, dry and weigh the cathode. The Pb can be determined separately as PbO_2 after dissolving the mixed deposit. To determine Zn in the residual electrolyte, add 2 *N* NaOH and redissolve the $Zn(OH)_2$ with the minimum of 2 *N* acetic acid (pH becomes $\simeq$ 4·6); heat to 40° C, electrolyse at 1·25 V for 25 min., wash with water and weigh the Zn.

An analysis can be completed in 2 hr. after the preparation of the soln. The results of a number of typical analyses are given. W. C. JOHNSON

2689. **Methods of sampling and testing boiler-water deposits.** British Standards Institution (B.S. 2455:1954, 45 pp.).—This specification, which applies to deposits formed in steam-generating equipment, covers definitions, and recommended methods of sampling and preparation of samples for physical and chemical analysis and of reporting results. Qual. tests for magnetic material, oil, organic matter, Fe, Cu, and the common anions are given, as well as full procedures for the quant. determination of water-soluble salts, oil, ignition loss, PO_4''', CO_3 (by the gas-evolution apparatus described and illustrated), SO_4'', Cl', Na, SiO_2 and Cu (colorimetrically for $\not>$ 100 μg, volumetrically for 0·005 to 0·05 g). Fe, Al, Ca and Mg are estimated in the filtrate and washings from the separation of sulphides during the determination of Cu; full procedures for phosphate and non-phosphate scales are given. Information likely to be obtained by microscope and X-ray examination of the sample is noted briefly. No tests are included for Zn, Ni and Cr, although it is appreciated that these may sometimes be present. Semi-micro or micro techniques may be needed for samples of small deposits of corrosion products in high-pressure boilers. W. J. BAKER

2690. **Treatment of water for land boilers.** British Standards Institution (B.S.2486:1954, 38 pp.).—The specification describes methods of treatment of water for land boilers and is supplementary to B.S.1170 "Treatment of water for marine boilers." Methods of testing are described and a table is given showing the relationship between methods of expressing hardness of water. W. J. BAKER

2691. **Determination of alkalis in coal ash.** R. Martinez Gayol (*Inst. Nac. Carbón Bol. Inf.*, 1954,

3 [13], 20–26).—Coal ash is treated with H_2SO_4 and HF to eliminate SiO_2. Hydroxides of tervalent metals are removed in the normal way by pptn. with aq. NH_3, and Ca and Mg are pptd. as oxalates in 85 per cent. acetic acid. The alkali metals are converted to sulphates, NH_3 is removed by heating, and the combined wt. of alkaline sulphates is calculated. The sulphate content is determined by pptn. as $BaSO_4$, and the Na_2O plus K_2O content is calculated. L. A. O'NEILL

2692. **Some aspects of the testing of clays for the pottery industry.** D. G. Beech and D. A. Holdridge (*Trans. Brit. Ceram. Soc.*, 1954, **53** [2], 103–133).—The importance of accurate sampling and the use of a sufficiently large sample is emphasised. A rapid method whereby routine checks of clay samples can be made is outlined. Three samples are taken: on one of them estimations of SiO_2 and Al_2O_3 are made; on the second the loss on ignition is determined; the third is used for alkali estimation. The first sample is fused with NaOH in a nickel crucible, the melt is extracted with H_2O, neutralised and made up to 250 ml. For the estimation of SiO_2, a portion of the solution is diluted, pH is adjusted, and SiO_2 is pptd. by the addition of ammonium molybdate and 8-hydroxyquinoline. The ppt. is filtered, washed, dried at 110° C and weighed. Alternatively, the SiO_2 can be determined volumetrically by pptg. it as the quinolate instead of as the hydroxyquinolate. The ppt. is dissolved in an excess of standard NaOH and back-titrated. From another portion of the solution Fe, Ni, Ti, etc., are pptd. by addition of NaOH. After filtering, the filtrate is neutralised with HCl, and made definitely acid. The last traces of Fe are kept in solution by reduction and formation of the ferrous *o*-phenanthroline complex. Al is then pptd. by addition of 8-hydroxyquinoline, followed by ammonium acetate to adjust the pH, the solution is heated to 70° C, and after it has settled, the ppt. is filtered, washed and dried at 110° C. The sample for alkali determination is gently ignited, heated with HF and H_2SO_4 and evaporated to dryness. The residue is dissolved in dil. H_2SO_4, diluted to 100 ml and the alkalis are estimated by means of a flame photometer. The degree of accuracy of these rapid methods compares favourably with the classical method. The mineralogical examination can be carried out by calculated rational analysis, by X-ray diffraction, by differential thermal analysis, and by electron microscopy. These methods are considered and discussed. Under physical testing, four methods of testing unfired clay are considered, *viz.*, modulus of rupture, particle size analysis, critical moisture content, and irreversible thermal expansion. A. J. MEE

2693. **Analysis of sands for glass manufacture.** C. L. de Marchi and G. C. Romagnoli (*Chim. e Ind.*, 1954, **36** [4], 253–263).—A scheme for the analysis of the fraction of $d > 2{\cdot}9$ separated from the sand by means of bromoform, involving the determination of Si, Fe, Al, Ti, Zr, Cr, Mn, Ca and Mg, is outlined. L. A. O'NEILL

See also Abstracts 2725, 2905.

3.—ORGANIC ANALYSIS

2694. **Volumetric determination of carbon and hydrogen in organic micro-combustion analysis.** A. Johansson (*Anal. Chem.*, 1954, **26** [7], 1183–1185).—A method for the volumetric determination of C and H in organic micro-combustion analysis is described and the apparatus is illustrated. The water formed during combustion is absorbed in a mixture of methanol and pyridine and determined by a Karl Fischer titration. The CO_2 is absorbed in an aq. soln. of NaOH and $BaCl_2$, and the pptd. barium carbonate is collected, converted to the iodate and determined iodimetrically. A comparison between the observed and calculated values for C and H for a wide variety of compounds is given. D. BAILEY

2695. **Adaptation of apparatus for the determination of carbon, hydrogen, oxygen and nitrogen, to the analysis of certain complex substances.** J. Garach and G. Valdener (*Chim. Anal.*, 1954, **36** [8], 211–214).—The classical combustion micro-method for C and H is modified by the use of a stream of air instead of oxygen, and the use of a separately heated Pyrex-glass tube containing Cu at 200° C outside the combustion tube, to decompose oxides of N. Oxygen is determined by pyrolysis in a current of N after formation of CO by means of C. The CO is oxidised with I_2O_5 on silica-gel and the CO_2 is absorbed and weighed. The increased weight of sample necessary, owing to the low O content of the material considered, makes necessary a slower pyrolysis than usual. The Dumas method for N is modified by passing evolved gases in a stream of CO_2 over a mixture of Cu and CuO at 750° C. A counter-current of CO_2 is used to displace the air admitted with the sample. Short descriptions and photographs of the three types of apparatus are given. E. J. H. BIRCH

2696. **The micro-analysis of fluorine-containing organic compounds. III. Determination of chlorine, bromine and iodine.** R. Belcher, A. M. G. Macdonald and A. J. Nutten (*Mikrochim. Acta*, 1954, [1], 104–116).—The micro-determination of Cl, Br and I in organic compounds containing F is described. The sample is heated with Na in a nickel bomb, and Cl is determined by titration. After addition of methyl red - methylene blue indicator, a saturated soln. of mercuric oxycyanide is added, and the soln. is titrated with 0·01 *N* H_2SO_4 to the neutral shade. To a similar vessel are added exactly the same amount of 0·01 *N* H_2SO_4, mercuric oxycyanide soln. and indicator, and the vol. is made up to approx. that of the test soln. This soln. is then titrated with 0·01 *N* NaCl to the same shade of indicator as the test soln. The amount of 0·01 *N* NaCl added is equiv. to the chloride in the test soln. Br is determined titrimetrically after oxidation to bromate by NaOCl. The iodide is determined after oxidation to iodate by Br. The method can be adapted to the simultaneous determination of Cl, Br and I, when present together in organic compounds containing F. A. J. MEE

2697. **Modification of the C-methyl determination as applied to very small samples.** C. F. Garbers, H. Schmid and P. Karrer (*Helv. Chim. Acta*, 1954, **37** [4], 1336–1338).—The Kuhn - Roth method of determining C-methyl is modified so that concn. of $\simeq$ 50 μg of acetic acid formed can be determined accurately by partition chromatography, thus enabling 0·2 to 0·4-mg samples to be analysed. The procedure is applicable also to analysis of the "B-acid" resulting from decomposition of vitamin B_{12}. W. J. BAKER

2698. **Rapid micro-analytical determination of acetyl and C-methyl groups.** W. Schöniger, H. Lieb and M. G. El Din Ibrahim (*Mikrochim. Acta*, 1954, [1], 96–103).—The method proposed depends on the determination of the acetic acid formed by hydrolysis of acetyl compounds or the oxidation of compounds containing the C-methyl group. The Parmas - Wagner modification of the micro-Kjeldahl apparatus is used. The acetic acid is quant. distilled off with steam within 10 min., and is then titrated with 0·1 *N* NaOH. The max. deviation from theory is ± 1 per cent.

A. J. MEE

2699. **Radiochemical studies on ultra-micro-quantities of organometallic compounds. I. On the determination of the composition of an organo-metallic compound in an ultra-micro amount.** T. Ishimori (*Bull. Chem. Soc. Japan*, 1954, **27** [3], 139–143).—The distribution of ultra-micro quantities of organometallic compounds between aq. and organic solvents (particularly $CHCl_3$) is examined theoretically. It is shown that a simple relationship exists between the partition ("extractability") and the number of organic ions associated with a metallic ion in a complex; by means of distribution measurements on radioactive isotopes of the cations, this number can be deduced. Certain possible defects in the procedure are discussed, and to examine them and the deductions from the theory, the constitution of lead (as thorium B) - dithizone (Dz) and lead - 8-hydroxyquinoline (Ox) complexes are investigated. The formulae $PbDz_2$ and $PbOx_2$ are confirmed, and a method for the examination of organometallic complexes of unknown compositions is proposed.

D. A. PANTONY

2700. **Establishing the composition of organic solvent - water mixtures in analytical precipitations.** R. E. Jentoft and R. J. Robinson (*Anal. Chem.*, 1954, **26** [7], 1156–1158).—An objective method is described for determining the ratio of organic solvent to water that results in the most complete separation, for the solvent considered, of a salt otherwise slightly soluble in water. The method, which is based upon mathematical derivation and involves graphical analysis of solubility data, is applied to a determination of the alcohol - water ratio having the max. solubility suppression effect upon KIO_4. The advantages of the method over experimental procedures are discussed.

D. BAILEY

2701. **A new spray reagent for paper chromatography of polyols and ketoses.** P. Godin (*Nature*, 1954, **174**, 134).—Paper chromatograms of polyols and sugars are sprayed with 1 per cent. ethanolic vanillin and 3 per cent. aq. $HClO_4$ (equal volumes mixed just before use), and heated at 85° C for 3 to 4 min. Glycerol, erythritol, xylitol, arabitol, adonitol, mannitol and sorbitol give pale blue or lilac spots that rapidly turn pale grey-blue, and sorbose and fructose give deep grey-green spots. Inositol, dihydroxyacetone, aldopentoses and aldohexoses give no colorations, except rhamnose which gives a brick-red spot. The min. quantities that can be identified are: ketohexoses, 5 μg; hexitols, 15 μg; rhamnose and pentitols, 20 μg; erythritol, 25 μg; and glycerol, 30 μg. Phenols and some indoles interfere, and bases and acids (particularly malonic and gluconic acids) must be removed.

C. E. SEARLE

2702. **Studies on ethylenic ketones. IV. Spectrophotometric estimation.** J.-M. Bonnier and G. de Gaudemaris (*Bull. Soc. Chim. France*, 1954, **21** [7–8], 997–1001).—Mesityl oxide ($\lambda_{max.}$ = 237 mμ, $\epsilon_{max.}$ = 12,100) in *iso*butyl methyl ketone ($\lambda_{max.}$ = 279 mμ, $\epsilon_{max.}$ = 23) is determined by measuring the extinction in ethanol at 237 mμ. For 1 to 8 mg of mesityl oxide per litre, Beer's law is obeyed. When < 6 per cent. of mesityl oxide is present, the extinction measurements are made against a blank containing the same proportion of pure *iso*butyl methyl ketone (or *iso*butyl methyl ketone containing a known amount of mesityl oxide) as the test solution. The error is less than 0·6 per cent. 4-Methylpentan-2-ol does not interfere. Dihydrophorone ($\lambda_{max.}$ = 240 mμ, $\epsilon_{max.}$ = 13,400) in di*iso*butyl ketone can be estimated in the same way.

E. HAYES

2703. **Formic acid as a reagent for alkaline permanganate. I. Potentiometric determination of formate.** I. M. Issa and R. M. Issa (*Anal. Chim. Acta*, 1954, **11** [2], 192–199).—Oxidation of formate with MnO_4' in 0·1 *N* or *N* NaOH at room temp. yields both MnO_2 and MnO_4''; equilibrium is reached slowly, and a potentiometric titration under these conditions gives no end-point inflection. At 80° C the same titration in 0·1 *N* NaOH yields MnO_2 as the only reduction product, equilibrium at the end-point being established in 5 to 10 min. if Ag^+ is present or in 8 to 10 min. if NaCl is present. Hence, 0·023 to 0·18 *N* formate in 0·1 *N* NaOH is titrated at 80° C with 0·066 *N* $KMnO_4$, the latter being 0·04 *N* with respect to $AgNO_3$. The max. error recorded is 0·13 per cent. A 0·0153 *N* formate soln. gives no inflection. A soln. of $KMnO_4$ (0·002 to 0·02 *M*) in 0·5 to 3·0 *N* NaOH can be titrated potentiometrically at 30° C with 0·0045 to 0·18 *N* formate; MnO_4'' is the only reduction product. When the same titration is conducted in the presence of 1 to 2 equiv. of Ba^{++}, the end-point inflection is greater, but the NaOH concn. must be 0·5 to 1·5 *N*. Equilibrium is established in ≃ 5 min., and the max. error is 0·1 per cent.

W. C. JOHNSON

2704. **Determination of lactic acid using ceric sulphate.** S. R. Elsden and Q. H. Gibson (*Biochem. J.*, 1954, **58** [1], 154–158).—Oxidation of lactic acid to acetaldehyde by $Ce(SO_4)_2$ is markedly affected by concn. of the oxidising agent and by temp. In the aeration method for determination of lactic acid, the initial concn. of $Ce(SO_4)_2$ should be 0·05 *N*, the temp. 50° to 60° C, and the rate of aeration should be 500 to 600 ml of air per min. for at least 45 min. A new steam-distillation method is described in which the acetaldehyde is removed by steam as fast as it is formed, and the $Ce(SO_4)_2$ is added gradually. The sample is mixed with 10 *N* H_2SO_4 and steam is passed through the mixture into a receiver containing aq. $NaHSO_3$. $Ce(SO_4)_2$ is added dropwise to the reaction mixture, and the acetaldehyde is determined by the usual iodimetric method. The apparatus for oxidation and distillation is described; the method is extremely rapid and one distillation requires only just more than 1 min.

J. N. ASHLEY

2705. **Determination of fumaric, malic and succinic acids in fermentation broths.** N. Lemjakov (*Anal. Chem.*, 1954, **26** [7], 1227–1228).—Fumaric acid is determined polarographically or by applying the mercurous fumarate method directly to the fermented substrate without extraction. After destroying the fumaric and malic acids with acid permanganate and evaporating to dryness in the presence of excess of $CaCO_3$, the succinic acid is determined by treatment with a known excess of

0·1 *N* $AgNO_3$ soln., filtration and titration of the excess of $AgNO_3$ in the filtrate. The total acids are determined by the silver nitrate method after evaporating the fermented substrate to dryness with $CaCO_3$; the malic acid is found by difference. D. BAILEY

2706. **Enzymic estimation of citric acid.** S. Dagley and E. A. Dawes (*Enzymologia*, 1953, **16** [4], 226–230).—From the culture medium of *Aerobacter aerogenes*, grown anaerobically with citrate as sole source of C, are prepared extracts that quantitatively convert citrate to pyruvate (in phosphate buffer, pH 7, at 37° C for 15 min.). Protein is pptd. with trichloroacetic acid, and the keto-acids in the supernatant soln. are converted to 2:4-dinitrophenylhydrazones, which are extracted with ethyl acetate and estimated on a Spekker absorptiometer. A linear relation is found between scale reading and citrate concentration in the incubation medium up to 6×10^{-4} *M* when fresh extract is used, and up to 4×10^{-4} *M* with stored frozen extract. No interference is found with a number of amino and other acids, but *cis*-aconitic and *iso*citric acid give rise to keto-acids. The method is applicable to bacterial culture media, urine, etc., provided that large amounts of keto-acids are not present. C. E. SEARLE

2707. **Quantitative method using paper chromatography for estimation of reducing oligosaccharides.** W. H. Wadman, G. J. Thomas and A. B. Pardee (*Anal. Chem.*, 1954, **26** [7], 1192–1195).—A method is described for rapid separation and estimation of oligosaccharides on a microgram scale. The sugars are allowed to react with N-(1-naphthyl)ethylenediamine [a solution of sugars and excess of reagent in triethylamine - ethanol - water (5:4:1) is spotted on paper and heated at 100° C for 30 min.] and are then separated by paper chromatography. The amount of each sugar is determined from the brightness of fluorescence of its deriv. under u.v. light. Application of the method to the study of hydrolysis of polysaccharides, metabolism of radioactive sugars and enzymatic hydrolysis of disaccharides is discussed. D. BAILEY

2708. **Detection of primary, secondary and tertiary amines with fused potassium thiocyanate.** F. Feigl and H. E. Feigl (*Mikrochim. Acta*, 1954, [1], 85–89).—When dry KCNS is fused with ammonium salts or with the hydrochlorides of primary, secondary or tertiary aliphatic or aromatic amines, H_2S is evolved. Less H_2S is produced with cyclic than with non-cyclic amines. This reaction can be used to detect these bases. A free base must first be converted into the hydrochloride. Organic substances that split off H_2O when heated must be absent, as such substances in amounts $>$ 500 μg give H_2S when fused with KCNS. To carry out the test, one drop of the solution of the amine in ether, chloroform or other organic solvent, plus one drop of HCl are evaporated to dryness at 110° C. The dry residue is mixed with an excess of well-dried KCNS and heated in an oil-bath to 200° to 250° C. The evolution of H_2S is detected by means of lead acetate paper. A. J. MEE

2709. **Complex compounds of silver as a basis for a simple titrimetric method of determining primary aliphatic amines, diamines and amino-acids.** H. Binder (*Angew. Chem.*, 1954, **66** [10], 268–271).—The complexes formed by Ag with compounds containing the amino group are often sufficiently stable for them to be used in argentimetric titrations of amino compounds with 0·1 *N* $AgNO_3$, with potassium chromate or carbonate or NaOH as end-point indicators. Although NH_3 can be quantitatively titrated, simple aliphatic primary amines, such as methylamine cannot, but cyclic aliphatic and aryl aliphatic amines can. The method can be applied to all diamines and amino-acids, provided the latter are first carefully neutralised. The reaction of $AgNO_3$ with hexamethylenediamine yields a compound of composition $[Ag(NH_2)_2(CH_2)_6]NO_3$, having m.p. 140° to 140·5° C. R. C. MURRAY

2710. **Determination of methyl nitrite formed in the preparation of nitromethane.** J. Décombe (*Bull. Soc. Chim. France*, 1954, **21** [5], 656–657).—Of the established methods for estimating ethyl and pentyl nitrites, only reduction by KI - H_2SO_4 is applicable to the more stable methyl nitrite, and for accurate results the solution must be dilute and the reaction conducted under CO_2 or N. Methanol (25 ml), the solution to be determined ($\simeq$ 2 per cent. with respect to methyl nitrite) (10 ml), H_2O (300 ml) and H_2SO_4 - H_2O (1 + 2) (30 ml) are placed in a 500-ml conical flask closed with a stopper accommodating the burette and gas delivery tubes. CO_2 is passed for a few min., 1 ml of 15 to 20 per cent. KI soln. is added rapidly, and, after shaking for 15 min. the liberated I is titrated with 0·1 *N* $Na_2S_2O_3$. By this means it is shown that the 53 per cent. yield of nitromethane obtained previously (*Bull. Soc. Chim. France*, 1953, **20**, 1038) is accompanied by 26 per cent. of methyl nitrite. E. J. H. BIRCH

2711. **Gravimetric determination of murexide.** J. H. Moser and M. B. Williams (*Anal. Chem.*, 1954, **26** [7], 1167–1168).—A direct method for the determination of murexide based upon the limited solubility of the Ca salt under prescribed conditions is described. The bulk of the murexide is pptd. by adding *M* $Ca(NO_3)_2$ soln. and adjusting the pH to 8·5 with 0·2 *N* NaOH; this is collected and weighed as calcium purpurate monohydrate. For high accuracy, the dye remaining in solution ($<$ 2 per cent.) is determined spectrophotometrically. The gravimetric assay of murexide provides an absolute method for the standardisation of a colorimetric analytical curve for this compound. D. BAILEY

2712. **Determination of ethylene dibromide and ethylene chlorobromide in air.** B. H. Kennett (*J. Agric. Food Chem.*, 1954, **2** [13], 691–692).—Allow the air sample to fill an evacuated 2-litre bulb having a stopcock of 8-mm bore. Add 50 ml of ethanol, shake the bulb for 15 sec. and transfer the contents to a 250-ml conical flask, rinse the bulb with 20 ml of ethanol and add the washings to the flask. Add 5 ml of *N* NaOH to the flask, reflux for 15 min., cool to $\not>$ 25° C, add 5 ml of 5 *N* HNO_3 and a known amount (10 to 20 ml) or 0·01 *N* $AgNO_3$ soln. Add 1 ml of ferric indicator (20 g of $FeSO_4$ and 10 ml of 5 *N* HNO_3 made up to 100 ml) and titrate with 0·01 *N* KCNS soln. Subtract the titre from that found for a blank determination without the halogen compound. Determine the bromide equiv. of the KCNS soln. by titrating it with 5 ml of 0·0100 *N* KBr soln. Mean recoveries were 99·5 $\pm$ 1·3 per cent. for ethylene dibromide and 99·4 $\pm$ 1·6 per cent. for ethylene chlorobromide. B. J. W.

2713. **A quantitative field test for quaternary ammonium compounds.** J. C. L. Resuggan and H. John (*Lab. Practice*, 1954, **3** [6], 231–232).—A rapid method for the routine quant. determination

of quaternary ammonium compounds is described. When 2 ml of a standard soln. of Na lauryl sulphate (Lorol) [0·408 g per litre when di-*n*-decyldimethylammonium bromide (Deciquam) is being estimated] are shaken with 3 ml of $CHCl_3$ and 5 ml of methylene blue soln., the blue complex formed separates into the $CHCl_3$ layer. The unknown soln. of quaternary ammonium compound is then added 0·5 ml at a time until the colours of the two layers match, owing to liberation of methylene blue from the $CHCl_3$-soluble complex. From the vol. of the unknown soln. required to reach this end-point the concn. of quaternary ammonium compounds can be determined. Reproducibility within 0·5 ml has been attained. N. M. WALLER

2714. **Polarographic determination of quaternary phosphonium salts.** E. L. Colichman (*Anal. Chem.*, 1954, **26** [7], 1204–1205).—Purified trimethylphenylphosphonium iodide (**I**) and tetra-*o*-tolylphosphonium iodide (**II**) are analysed polarographically in 95 per cent. ethanol with tetraethylammonium iodide (0·05 or 0·1 *M*) as supporting electrolyte, and either methylcellulose or trimethyltetradecylammonium bromide as maximum suppressor. Polarographic data are as follows: **I**, $E_{\frac{1}{2}}$ 2·18 to 2·33, i_d/C 1·70 to 1·90, a double 1-electron reduction; **II**, $E_{\frac{1}{2}}$ 2·35 to 2·40, i_d/C 3·60 to 3·95, a single 2-electron process. D. A. PANTONY

2715. **Polarographic determination of phthalaldehyde.** N. H. Furman and D. R. Norton (*Anal. Chem.*, 1954, **26** [7], 1111–1115).—A polarographic investigation of phthalaldehyde is described and a method for the determination of low concn. of this compound is based on these studies. Phthalaldehyde is reduced at the dropping-mercury electrode, to give 2 waves. The half-wave potentials of both waves become more negative with increasing pH. The reduction current constants of the first wave do not vary for pH values between 3 and 9, but increase for values between 9 and 12, whilst the constants for the second wave increase between pH 3 and 9 and decrease between pH 9 and 12. Limiting currents are controlled by the equilibria between reducible and non-reducible forms of phthalaldehyde, by their rates of diffusion and by their rates of reaction. The reduction current of each wave in buffered solutions between pH 3 and 12 is proportional to the concn. of the aldehyde in the range of 0·40 to 2·00 millimoles. The effects of change in buffer capacity, solvent concn., mercury pressure and temp. are discussed. The polarographic determination of low concn. of the aldehyde is based upon the linear relationship between reduction current and concn. The method is reliable and accurate (average deviation < 2 per cent.). D. BAILEY

2716. **Quantitative estimation of aromatic nitro compounds.** E. Wolthuis, S. Kolk and L. Schaap (*Anal. Chem.*, 1954, **26** [7], 1238–1240).—A method is described for the quant. determination of aromatic nitro compounds. The substance is reduced by zinc dust and HCl in acetic acid, and, after removing the excess of Zn, the resulting amine is determined volumetrically with 0·1 *N* $NaNO_2$. Good results are obtained for mono-nitro compounds except the iodonitrobenzenes. A qual. test for aromatic nitro compounds based on this procedure is discussed. D. BAILEY

2717. **Titration of aromatic and aliphatic amine picrates in non-aqueous solution.** J. R. Clark and S. M. Wang (*Anal. Chem.*, 1954, **26** [7], 1230).—A rapid method for the determination of the milli-equiv. wt. of amine picrates by direct titration with $HClO_4$ in acetic acid is described. By using methyl violet as indicator, a reasonably sharp end-point, unaffected by the yellow coloration of the picrates, is attained. The method is useful for both aromatic and aliphatic primary, secondary and tertiary amines. D. BAILEY

2718. **Chromatographic separation of *p*-phenylazophenacyl esters on silicic acid.** R. M. Ikeda, A. D. Webb and R. E. Kepner (*Anal. Chem.*, 1954, **26** [7], 1228–1229).—Chromatographic purification and separation of *p*-phenylazophenacyl esters on silicic acid is described. The following m.p. of *p*-phenylazophenacyl esters are reported: acetate, m.p. 125° to 127°C; propionate, m.p. 104° to 105°C; butyrate, m.p. 98° to 99°C; *iso*butyrate, m.p. 101·5° to 102°C; valerate, m.p. 69·5° to 70°C; caproate, m.p. 83° to 84°C; caprylate, m.p. 83° to 83·5°C; caprate, m.p. 84·5° to 85·5°C; laurate, m.p. 87° to 87·5°C; myristate, m.p. 92° to 93°C; palmitate, m.p. 95° to 96°C; stearate, m.p. 99° to 100°C. The result of the separation of 19 pairs of esters of 12 aliphatic acids and 3 pairs involving esters and reagent are reported. For the separation of esters of low molecular weight, the developing solvent is benzene - Skellysolve B (3 + 1) and for esters of high molecular weight the ratio is 1 to 1. The separation of these esters is superior to that of *p*-phenylphenacyl esters. D. BAILEY

2719. **A new colorimetric method for the determination of salicylhydroxamic acid and its analogues.** J. Venulet (*Bull. Acad. Polon. Sci.*, 1954, **2** [4], 195–198).—A reagent (1 ml) made by dissolving 0·25 g of sodium or ammonium vanadate in 20 ml of *N* H_2SO_4 and making up to 100 ml is added to 2 ml of the solution containing a hydroxamic acid, and 2 ml of water are added. After 2 to 3 min. the violet colour reaches a stable max. and is measured on a Pulfrich photometer at 610 mμ. The colour is obtained in acid or neutral solution, and the Beer - Lambert law is obeyed over the range 10 to 20 μg per ml. Only those hydroxamic acids with a hydroxyl group ortho to the hydroxamic group in an aromatic nucleus give the violet colour. E. J. H. BIRCH

2720. **N-Oxides of the pyridine series and their application to the paper-chromatographic analysis of mixtures of pyridine bases.** D. Jerchel and W. Jacobs (*Angew. Chem.*, 1954, **66** [11], 298).—A mixture of alkylpyridine bases is dissolved in glacial acetic acid and oxidised by heating with 30 per cent. hydrogen peroxide for 10 min. under reflux, and the resulting liquid is chromatographed on paper by means of butanol - formic acid - water (75:15:10) as eluting soln.; the stains are developed with 0·005 per cent. alcoholic acridine, which gives dark stains in the presence, and yellow stains in the absence, of N-oxides of pyridine, alkylpyridines or quinoline. A table of R_F values is given, and a simple method of reading the R_F value for a stain from its position by means of a "proportional protractor" is described. R. C. MURRAY

2721. **Quantitative determination of pyridine bases by chromatographic adsorption.** A. Waksmundzki and J. Ościk (*Roczn. Chem.*, 1954, **28** [2], 239–249).—Chromatographic determination of pyridine bases in aq. solution by the frontal analysis method and Tiselius - Claesson apparatus is described. The

accuracy of the method is within 4 to 6 per cent.; it depends on the sharpness of front of each substance in the eluate, which can be improved by applying in the column a system of progressively narrowing filters. 3-Picoline and 2:6-lutidine were well differentiated. The method can be used for determining the composition of mixtures and for testing the purity of commercial compounds.

S. K. LACHOWICZ

2722. **Colour standards [in the petroleum industry].** G. J. Chamberlin (*Inst. Petrol. Rev.*, 1954, **8**, 121–122).—A re-statement is given of the meanings of the figures assigned to a number of colour scales used in the petroleum industry. The I.P. standard glasses are defined in terms of Lovibond colours (yellow 510 and red 200), the A.S.T.M. and Indian colour scales for lubricating oils are shown in relation to Lovibond analyses, and the Redwood scale is compared with I.P. markings (1924).

B. J. W.

2723. **A rapid method for determining the char value of kerosine.** A. R. Javes and C. Liddell (*J. Inst. Petrol.*, 1954, **40**, 170–174).—The sample is rapidly distilled under nitrogen at atmospheric pressure to a 7 per cent. residue. The residue is burned in a modified Institute of Petroleum burning-test lamp for 5 hr., and the char is separated from the end of the wick, extracted with light petroleum and weighed. Results obtained in 8 hr. correlate with those by the 27-hr. I.P. Standard char- value test IP 10/53 (T).

J. G. KING

2724. **[Review of coal tar technology.] Analytical chemistry.** The Coal Tar Research Association (*Rev. Coal Tar Technol.*, 1953, **5** [2], 142–150).—A review of developments in methods of analysis applied to coal tar and its products during the period July to December, 1953, with 78 references.

N. E.

2725. **Determination of titanium in textiles.** Anon. (*Shirley Inst. Test Leaflet*, 1954, No. Chem. 21).—Organic matter is destroyed by wet- or dry-ashing. In the volumetric procedure, which can deal with larger amounts of Ti and is more accurate then the colorimetric method although it is more subject to interferences, the ash solution is passed through a Jones reductor containing zinc amalgam into a ferric alum solution, which is then titrated against ceric ammonium sulphate. Modifications in the presence of Fe, Sb, Cu, Sn and Cr are described. In the colorimetric method, the ash solution, acidified to 2 to 3 *N* with H_2SO_4 and containing 2 per cent. sodium sulphate, is treated with H_2O_2. The amount of Ti corresponding to the difference in optical density between this solution and a blank is determined by reference to a calibration graph. Modifications are given for when Cr, Cu, Fe and PO_4''' are present.

A. M. SPRATT

2726. **Analytical methods for the detection of impurities in the [cellulose] acetate textile industry.** R. H. McKinlay (*Fibres*, 1954, **15** [2], 39–42).—The detection and quant. determination of traces of Fe, Cu, Mn, Zn, Ni, Cr, Ca, Al, SiO_2, Cl and S in raw materials, intermediates or finished products are reviewed. The organic matter is first removed by ashing at a low temp. or by a wet oxidation process with conc. H_2SO_4 and conc. HNO_3. Many tests are colorimetric, and use is made of the Spekker absorptiometer, Grubb - Parsons i.r. spectrometer, Hilger u.v. spectrometer and electrolytic apparatus for metal separation. The use of the flame photometer is being developed.

A. M. SPRATT

2727. **The estimation of total sulphur in viscose.** B. Philipp (*Faserforsch. u. Textiltechnik.*, 1954, **5** [5], 210–211).—As the total S (total of sulphide, thiocarbonate and xanthate S) shown by potentiometric methods is as much as 50 per cent. lower than that shown by the usual works method (oxidation with H_2O_2 and alkalimetric titration), an investigation was made of methods of determining the total S in viscose. The following methods are used and compared: (*i*) the usual works method, by oxidation with H_2O_2, estimation of the difference in alkali content before and after oxidation and calculation of the H_2SO_4 produced, (*ii*) gravimetric estimation as $BaSO_4$ (*a*) after oxidation with Br in alkaline solution, and (*b*) after H_2O_2 oxidation in alkaline solution, (*iii*) gas estimation of total CS_2 and H_2S by decomposing 4 to 5 g of viscose with 50 ml of 2 *N* HCl, absorbing the emitted gas successively in 5 per cent. $CdSO_4$ acetate-buffered solution and in 10 per cent. alcoholic KOH, and estimating the S in the residue by Br oxidation followed by precipitation as $BaSO_4$. Method (*ii a*) is considered to be the most accurate and method (*iii*) gives results in closest agreement with those of (*ii a*). Method (*ii b*) gives results in fair agreement with those of (*ii a*) and is quicker than the hypobromite oxidation method, but it is not considered suitable as a standard method because of high false values caused by sulphate present in the H_2O_2; complications in oxidising the thiocarbonate with H_2O_2 are also possible. The results of method (*i*) are too high, being $\simeq$ 16 per cent. higher than those of (*ii*) and (*iii*). The reasons for this are discussed, and revision of works methods for determining S in viscose is advised.

H. L. WHITEHEAD

2728. **Simple method of comparing breaking loads of two yarns.** S. L. Anderson (*J. Text. Inst.*, 1954, **45**, T472–T479).—A simple method is described, requiring no strength-testing machine, for deciding which of two yarns is the stronger. The yarns are broken by pulling by hand, and the number of breaks in each yarn is noted and treated by the statistical method of sequential analysis. Only an approx. value of the coeff. of variation of breaking load need be known.

L. VALENTINE

2729. **Determination of mercury in pulp and paper.** O. T. Carlson and P. O. Bethge (*Svensk Papperstidning*, 1954, **57**, 405–408).—The sample is completely oxidised by boiling with a mixture of $HClO_4$ and H_2SO_4. When oxidation is complete, the condensate and the oxidising mixture are combined and after dilution with water, brought to pH 1 with aq. NH_3. Oxidising substances still present are reduced with hydroxylamine sulphate. The mercury content of the solution obtained is determined colorimetrically with dithizone according to the reversion process of Irving *et al.* The method gives good reproducibility and the amount of Hg found agrees well with the amount originally added.

S. V. SERGEANT

2730. **Quantitative determination of copper in paper.** P. V. Shumilov and A. Ya. Kul'mer (*J. Appl. Chem., U.S.S.R.*, 1954, **27** [1], 109–111).—Quant. determination of Cu in paper is rendered difficult by the presence of Fe at concn. exceeding 3 to 5 times the concn. of Cu ($\simeq$ 0·005 per cent.). In experiments with several masking agents, results are best by adding NH_4F, which forms colourless

[FeF]$^{'''}$ ions that do not react with I$'$ in weak acid solutions. A detailed experimental procedure is described for the determination of Cu in paper ash by titrating I liberated by Cu with $Na_2S_2O_3$ solution; max. error is ± 1·3 per cent. S. K. LACHOWICZ

2731. **Polarographic determination of chlorine in vinyl chloride co-polymers.** M. Pražák, J. Benc and Z. Bartušek (*Chem. Průmysl*, 1953, **3**, 297–298).—Dried-out polymer (0·015 g) is wrapped in cigarette paper and burned in a closed flask between two platinum electrodes by means of a thin glowing platinum wire. The flask contains 50 ml of H_2O, and O is introduced for 5 min. during the ignition process. The flask is shaken to absorb the combustion gases. Ten ml of the soln. are pipetted into a small beaker, 10 ml 0·2 *N* H_2SO_4 and ≃ 0·01 g of water-free $Na_2S_2O_3$ are added, the liquid is stirred and after 5 min. transferred to a polarographic cup that has been previously rinsed with the soln. and the bottom covered with Hg. The potential used for the measurement is + 0·130 to + 0·135 mV on the drop. From the determined normality of the soln. the amount of Cl in the sample is calculated. The method is suitable for polymers containing up to 73 per cent. of Cl. The accuracy of the method is ± 0·5 per cent.
CHEM. ABSTR.

2732. **Flame-photometric determination of rubber solids deposited on cords and fabrics.** H. E. Todd and H. M. Tramutt (*Anal. Chem.*, 1954, **26** [7], 1137–1140).—The adhesion between fibres and rubber in the production of tyres, belts and hose depends upon the amount of rubber solids deposited on the fibres during a pre-treatment with suitable latex-based formulations. The existing methods (chemical and mechanical) for determining the percentage of latex solids picked up during pre-treatment are reviewed, and a method based on flame photometry is presented. The method, which is applicable to all cords and fabrics and any latex formulation containing Na, is based upon the variation of the Na content in the system and its evaluation on cotton, rayon, nylon, Fiberglas and Dacron by use of natural rubber, GR-S and other special latexes. Satisfactory speed and accuracy can be achieved. D. BAILEY

2733. **Method for copper and iron in tanning materials.** Anon. (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [4], 238–243). The provisional method of the American Leather Chemists' Association for determination of Cu and Fe in liquid and solid tanning extracts, and raw and spent tannery liquors, by use of $KMnO_4$ and KCNS for Fe, and K ethylxanthate for Cu is detailed. The sample is dried, and ashed at temp. below 600° C, and the ash is dissolved in HCl. Aliquots of this solution are used for the determination. B. R. HAZEL

2734. **Analysis of leather finishing materials.** Anon. (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [4], 258–262).—Methods of analysis of finishing material for use on leather are given for total solids and ash, cellulose nitrate, flexibility for adhesion of finish and tackiness. B. R. HAZEL

2735. **Method for analysis of beamhouse liquors.** Anon. (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [4], 243–258).—Methods are given in detail for sampling soak waters, lime liquors and bate waters, and for determination of N, NH_3, caustic alkalinity, Ca, Cl$'$, S$''$, SO_4'' and pH. B. R. HAZEL

See also Abstracts 2821, 2857.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE
Blood, Bile, Urine, etc.

2736. **Haemoglobin standard.** J. C. F. Poole (*Lancet*, 1954, **ii** [3], 116–117).—The most reliable value for the iron content of haemoglobin is 0·340 per cent. (Bernhart and Skeggs, *J. Biol. Chem.*, 1943, **147**, 19). The British Standards Institution Haldane colour standard on this basis is equivalent to 14·5 and not 14·8 g of haemoglobin per 100 ml of blood. C. E. SEARLE

2737. **An improved method for multiple rapid determinations of arterial blood pH.** D. A. Holaday (*J. Lab. Clin. Med.*, 1954, **44** [1], 149–159).—Details are given of the construction and operation of an electrostatically shielded air-bath and a modified glass electrode assembly for use with a conventional pH meter. With the apparatus described, pH values can be determined at body temp. to within 0·02 units of pH on about 2 ml of arterial blood at two-minute intervals over an extended period of time. Problems relevant to the determination of pH values on biological fluids are discussed. W. H. C. SHAW

2738. **The use of the Cavett apparatus in micro-analysis.** R. F. Milton and W. D. Duffield (*Lab. Practice*, 1954, **3** [8], 318–323).—The Cavett micro-diffusion apparatus (*J. Lab. Clin. Med.*, 1937, **23**, 543) consists of a 50-ml Erlenmeyer flask having a ground-in stopper carrying a small cup in which the sample (0·1 to 0·5 ml) and reagent(s) are placed. After putting the absorption liquid into the flask the stopper is inserted and held tight by spring clips fastened to lugs on the flask and stopper. There is more space for active diffusion than in the Conway flask, and the apparatus can be heated without the stopper being loosened. In estimations of alcohol (0·1 mg) in blood and urine, reproducible results from various sources showed deviations of ≯ 5 per cent. Procedures for the determination of each of the following constituents in clinical samples (mainly by standard methods of gas liberation and absorption) are given: NH_3, total N, urea (by hydrolysis with urease and absorption of NH_3 in H_3BO_3), nitrate and nitrite (in soils), halogens, CO_2, glucose [by fermentation with yeast and absorption of CO_2 in 0·01 *N* $Ba(OH)_2$], acetone (by fixation as bisulphite and titration with I), alcohol, chloroform (by diffusion into toluene and colorimetric estimation with pyridine and 20 per cent. NaOH), and CO. Results obtained with the Cavett flask are compared with those obtained by the usual micro-methods.
W. J. BAKER

2739. **Flame-photometric micro-determination of sodium, potassium, and calcium in serum. Effect of organic solvents.** G. R. Kingsley and R. R. Schaffert (*J. Biol. Chem.*, 1954, **206** [2], 807–815).—The use of organic solvents extends the sensitivity of the flame photometer in the direct determination of Na, K and Ca and makes practicable the analysis of very small biological specimens. The use of an acetone - glacial acetic acid - water mixture (27 : 9 : 4) with the addition of 0·02 per cent. of Sterox SE (a non-ionic wetting agent) to prevent adsorption of Ca on the glass is advised. Accurate results for Na, K and Ca of sera and urine are reported for specimens as small as 0·000005 ml, 0·0005 ml and 0·0006 ml, respectively, as determined by dilution.
N. E.

2740. **The estimation of copper, lead and zinc in blood plasma by means of polarography and cation exchange.** G. Kahle and E. Reif (*Biochem. Z.*, 1954, **325** [5], 380–388).—Copper, lead and zinc in plasma can be estimated polarographically after a preliminary separation by (*i*) moist ashing of the plasma with subsequent removal of the phosphate by the cation-exchange resin Wofatit or (*ii*) direct separation of the elements on cation exchangers. The first method has been extensively investigated and details are given of the time- and temperature-controlled ashing technique on serum or plasma (10 ml), 65 per cent. HNO_3 (10 ml), 70 per cent. $HClO_4$ (5 ml) and conc. HCl, sp. gr. 1·19 (1 ml). The validity of the method has been proved by estimation of 10 μg of each of the three elements in (*a*) a saline solution of inorganic salts at concentrations similar to those in blood, (*b*) as in (*a*) with subsequent moist ashing and removal of phosphate and (*c*) in plasma. No indications were found of any loss in either ashing or removal of phosphate. The ashing method has been compared with the direct cation-exchange method. Full details are given of the technique and the polarographic apparatus. G. W. CAMBRIDGE

2741. **Simplified estimation of calcium in biological materials by flame photometry.** J. G. Llaurado (*J. Clin. Pathol.*, 1954, **7** [2], 110–115).—The Ca is pptd. as oxalate and the ppt. is heated to convert it to $CaCO_3$ to avoid interference by the oxalate ion in the flame photometry. To 1 ml of serum in a 15-ml Pyrex-glass centrifuge tube, add 1 ml of water and 1 ml of 4 per cent. w/v ammonium oxalate, mix and set aside for 30 min. Add 4 ml of water, mix and centrifuge at 2500 r.p.m. for 10 to 15 min. Decant the supernatant soln. and leave the tube inverted over filter-paper for 5 min. Place the tube overnight in a furnace at 300° to 400° C. Dissolve the residue in 5 ml of 0·2 *N* HCl, immersing in boiling water for 2 min., cool and dilute to 10 ml with water. Prepare a stock soln. of Ca by dissolving 15·2 g of pure $CaCO_3$ in 250 ml of *N* HCl and diluting to 1 litre with water (1 ml ≡ 5 mg of Ca), and dilute aliquots to make standards containing 10, 20, 30 and 40 p.p.m. of Ca. Spray the test soln. and standards in the flame photometer using an interference filter with peak at 620 mμ and a special didymium glass to cut out sodium light. H. F. W. KIRKPATRICK

2742. **Flame-photometric determination of electrolytes in tissue and of calcium in serum.** J. R. Denson (*J. Biol. Chem.*, 1954, **209** [1], 233–240).—A flame-photometric method is described for the simultaneous determination of Ca, Mg, Na and K in tissues. The sample (2 g) is weighed, dried at 90° C *in vacuo* for 16 hr and re-weighed to determine the moisture content; it is then extracted with ether (15 ml) at 0° C for 2 hr, again with ether (10 ml), and dried to enable the fat-free wt. to be determined. The de-fatted material is ashed in a furnace at 500° C for 6 hr., the ash is dissolved in conc. HCl (1 ml) and the solution is evaporated to dryness. The residue is dissolved in 0·1 *N* HCl (10 ml) and the solution is poured through a column (10 cm long; capacity ≃ 3·5 ml) of Amberlite IR-105 (to remove phosphate), the column is washed with water (15 ml), and the salts are eluted with 6 *N* HCl. The eluate is evaporated to dryness, the residue is dissolved in 0·1 *N* HCl, and the sample is then analysed for Ca, Mg, Na and K by the flame-spectrophotometric method by use of a hydrogen burner. The wavelengths used are 589 (Na), 789 (K), 554 (Ca) and 285·2 mμ (Mg). A correction is applied to the Ca reading because of the extra Ca eluted from the column. For determination of K and Na, the solution is diluted (1 + 10) and (1 + 100), respectively. The amount of each element is then ascertained from standard graphs. Fe, if present, is removed after ashing by cupferron. Serum is analysed by diluting the soln. (1 + 10) and passing it through the column. The eluate (in 6 *N* HCl) is then treated as above. Mg cannot be determined in serum in this manner because its concn. is too low. J. N. ASHLEY

2743. **Determination of iron in small amounts of serum and whole blood with the use of thiocyanate.** H. W. Josephs (*J. Lab. Clin. Med.*, 1954, **44** [1], 63–74).—The thiocyanate method is adapted for use with 0·5 to 1·0 ml of serum or 0·1 ml of whole blood. Iron complexes in the sample are broken down by preliminary acid digestion, proteins are pptd. with trichloroacetic acid and, after centrifuging, the Fe in the supernatant liquid is oxidised with dilute HNO_3. The colour developed with NH_4CNS is extracted into ethyl acetate and measured on a filter (540) photometer. The iron levels found in various types of anaemia are discussed. W. H. C. SHAW

2744. **The determination of alcohol in blood.** S. Wehrli (*Mitt. Lebensm. Hyg., Bern*, 1954, **45** [2], 123–141).—The procedure used by the Medico-Legal Institute of Zurich for the determination of alcohol content of blood is described. The importance to the analyst of obtaining full medical information is stressed. The tests are divided into the qual. identification and quant. estimation of the alcohol. The following tests are always performed. *Qualitative*—(*a*) Formation of ethyl benzoate, (*b*) iodoform, (*c*) Tollens reaction for reducing materials and (*d*) a test for ketones. *Quantitative*—Ten g of blood are distilled in a special apparatus and the alcohol in the distillate is determined by two methods: oxidation with chromic acid and determination of refractive index. Only when the results by the two methods agree can the presence of alcohol be inferred. The effects of the following materials in the test sample have been studied and are discussed: ether, acetone, ammonia, amines, formaldehyde, methanol, organic solvents, carbon monoxide, drugs, chloral hydrate and disinfectants (from cleansing of the skin). A comparison with clinical observations shows that trained people can always recognise the symptoms of intoxication if the alcohol content of the blood exceeds a certain minimum. P. S. STROSS

2745. **The application of Goldberg's evaluation methods in alcoholics.** G. Dotzauer, K. Johannsmeier and H. Redetzki (*Dtsch. Z. ges. gerichtl. Med.*, 1953, **42**, 247–253).—Goldberg has worked out mathematical formulae, based on experimental evidence, to relate the time of absorption of alcohol to its concn. in serum. The experiments are repeated and it is shown that Goldberg's formulae cannot, as yet, satisfactorily reflect the complex physiological reactions of absorption, diffusion and levels of concn. of alcohol in blood. CHEM. ABSTR.

2746. **Analytical determination of alcohol in the breath.** K. Grosskopf (*Angew. Chem.*, 1954, **66** [11], 295–297).—The ratio of the alcohol concentration in the blood (in mg per 100 ml) to that in the

alveolar air (in mg per litre), *viz.*, the Ostwald solubility coeff., is approximately constant for up to 0·25 per cent. of alcohol in the blood, the limit normally met with, and is 630 at 20°, 313 at 30°, 208 at 37°, and 177 at 40° C, so that preliminary estimates of blood alcohol concentrations can be made from determinations of the alcohol in the breath. This is done by emptying the lungs at a flow rate of 1 litre per min. through a tube containing H_2SO_4 - CrO_3 mixture absorbed in silica gel having a resistance equal to 35 to 55 mm H_2O, and measuring the length of the green zone of Cr_2O_3 produced to give an index of the alcohol content of the breath. A calibration curve is given, and the sensitivity and value of the test are discussed.

R. C. MURRAY

2747. **A new tablet test for bilirubin in urine.** J. A. Tallack and S. Sherlock (*Brit. Med. J.*, 1954, **ii**, 212–213).—The tablet contains a stable diazo dye (*p*-nitrophenyldiazonium toluene-*p*-sulphonate), sulphosalicylic acid, $NaHCO_3$ and boric acid. Place 5 drops of urine on a test mat 19 mm square and 2 mm deep composed of a mixture of asbestos and cellulose fibres. Place the tablet in the centre of the moistened area and allow 2 drops of water to flow on the tablet with an interval of a few sec. between the drops. Record the colour that develops on the mat within 30 sec., ignoring any colour developed after this period. If the urine contains bilirubin, the mat around the tablet turns purple, the intensity of colour being roughly proportional to concn. of bilirubin. The test will detect between 0·1 and 0·15 mg of bilirubin per 100 ml of urine and is similar in sensitivity to Fouchet's test. The iodine method was found to be too insensitive for routine use in detecting small amounts of bilirubin.

H. F. W. KIRKPATRICK

2748. **Spectrophotometric study of some oxidation products of bilirubin.** B. Zak, N. Moss, A. J. Boyle and A. Zlatkis (*Anal. Chem.*, 1954, **26** [7], 1220–1222).—The determination of bilirubin by spectrophotometric analysis of its oxidation products with a sulphuric acid - acetic acid colour reagent and a perchloric acid colour reagent is described. The difference in spectra of bilirubin in the H_2SO_4 - acetic acid solution of $FeCl_3$ before and after dilution with water is caused by oxidation (dehydrogenation) to bilatriene with increased conjugation in one instance and oxidation of bilirubin with the $HClO_4$ solution of $FeCl_3$ to form biladienes in the other. In physiological media such as serum, the former reagent is used in the absence of cholesterol, and the latter reagent when tryptophan is absent. Recovery of bilirubin added to serum is good.

D. BAILEY

2749. **Quantitative determination of glucose by paper-partition chromatography.** S. Baar (*Biochem. J.*, 1954, **58** [1], 175–176).—The solution containing glucose is poured on to filter-paper, the chromatograms are treated with aniline hydrogen phthalate in *iso*propanol, the papers are heated at 115° ± 1° C for 15 min., and the stained areas are cut out and extracted with acetic acid at room temp. The extract is examined spectrophotometrically at 480 mμ, and the amount of glucose is ascertained from a standard graph. Although the method (which is very sensitive) is a general one lacking specificity, it is well suited to determination of glucose on chromatograms, as lack of specificity is replaced by good chromatographic separation.

J. N. ASHLEY

2750. **A method for the estimation of the neutral hexose of protein in blood-serum.** C. v. Holt (*Klin. Wochschr.*, 1954, **32** [27–28], 661–662).—The proteins of serum (0·1 ml) are precipitated by treatment with absolute alcohol (4 ml) in a centrifuge tube. After decanting the supernatant liquid, the residue is washed with alcohol (4 ml). The residue is taken up in 0·9 per cent. of NaCl (3·9 ml) and 1 ml is transferred to a tube containing ice-cold anthrone soln. (0·2 per cent. in 78 to 79 per cent. H_2SO_4, sp. gr. 1·717) (10 ml). After mixing and heating on a bath of boiling water for exactly 5 min., the reaction is stopped by placing the tube in ice. Photometric estimation of the extinction of the green colour is made at 623 mμ or 578 mμ (1-cm thickness). A standard calibration curve is made by using equal amounts of mannose and galactose (1 ml of soln. containing 25, 50 or 75 μg per ml). Error is stated to be ± 1 per cent.

G. W. CAMBRIDGE

2751. **Simultaneous determination of sucrose and inulin in biologic[al] fluids.** M. K. Young and J. F. Prudden (*J. Lab. Clin. Med.*, 1954, **44** [1], 160–165).—The anthrone procedure for inulin described previously (*Proc. Soc. Exp. Biol. Med.*, 1952, **80**, 771) is developed for the accurate determination of sucrose and the simultaneous determination of sucrose and inulin. The two substances are determined in plasma and urine from the optical densities at 630 mμ obtained with anthrone before and after invertase treatment and after subsequent digestion with NaOH. The accuracy attained and the applications of the technique are discussed.

W. H. C. SHAW

2752. **The use of substances depressing antithrombin activity in the assay of prothrombin.** P. Fantl (*Biochem. J.*, 1954, **57** [3], 416–421).—Phenols depress the antithrombic activity of serum. This property, as well as the accelerating effect of phenols on the thrombin - fibrinogen reaction, is utilised in a prothrombin assay which is based on that of Warner *et al.* (*Brit. Abstr. A*, 1936, 1402). One of the essential differences between the two methods is that the gum acacia in the older method is replaced by catechol in the proposed method. The plasma need not be defibrinated and fibrinogen can be replaced by human Ba-plasma; rabbit Ba-plasma is less reactive towards thrombin. Citrated plasma should be used, and for normal prothrombin concn. it should be diluted 120 times. Macro and micro forms of the method, which is applicable to blood, plasma and serum of man and other mammals, are described.

J. N. ASHLEY

2753. **The determination of cyanide in biologic[al] fluids by micro-diffusion analysis.** M. Feldstein and N. C. Klendshoj (*J. Lab. Clin. Med.*, 1954, **44** [1], 166–170).—A rapid method requiring a standard Conway diffusion dish is described for materials containing 0·1 μg or more of cyanide in 4 ml of sample. The cyanide level of normal blood plasma is shown to vary from zero to 14 μg per cent.; various factors affecting the levels found are discussed.

Procedure—Place a 3 to 5-ml aliquot of biological fluid or of an aqueous tissue homogenate (diluted 1 + 4) in the outer chamber of a Conway diffusion dish and 1·0 or 2·0 ml of 0·1 *N* NaOH in the central chamber. Add 1 ml of 10 per cent. H_2SO_4 to the outer chamber, immediately place the cover in position and mix by rotating gently. After 2 hr., remove an aliquot of the NaOH containing 0·1 to 2·0 μg of cyanide and complete the colorimetric

determination of cyanide by the pyridine - pyrazolone method of Boxer and Rickards (*Brit. Abstr. C*, 1951, 267). W. H. C. SHAW

2754. **A micro-method for the estimation of *p*-aminohippuric acid in capillary blood.** K. H. Kimbel (*Klin. Wochschr.*, 1954, **32** [23–24], 566).—Blood (0·2 ml) is freed from proteins by the Hagedorn - Jensen method [0·45 per cent. aq. $ZnSO_4$ (5 ml) and 0·1 *N* NaOH (1 ml). After centrifugation, the supernatant fluid is transferred to a test tube and 2 *N* HCl (0·5 ml) followed by freshly prepared 0·1 per cent. $NaNO_2$ (0·5 ml) is added. After shaking and setting aside for 5 min., 2 per cent. sulphamic acid soln. (0·5 ml) is added and 5 min. later 1 per cent. N-ethyl 1-naphthylamine hydrochloride soln. (0·5 ml) is added. After 30 min. the red colour is determined at 530 mμ. Calibration is carried out over a range of 1 to 10 mg per cent. of *p*-aminohippuric acid. G. W. CAMBRIDGE

2755. **Neuramine acid, its occurrence and its estimation in serum.** P. Böhm, St. Dauber and L. Baumeister (*Klin. Wochschr.*, 1954, **32** [13–14], 289–294).—Neuramine acid (*Hoppe-Seyl, Z.*, 1941, **268**, 50) can be determined as follows. Serum (0·05 ml) is mixed with dist. H_2O (1 ml) in a centrifuge tube (graduated at 2 ml and fitted with a ground-glass stopper). The proteins are pptd. by 10 per cent. trichloroacetic acid (1 ml), and, after centrifuging, the residue is suspended in 5 per cent. trichloroacetic acid (2 ml), centrifuged again and then suspended in H_2O (0·5 ml). To this is added Bial's orcinol - $FeCl_3$ reagent (1 ml) and the mixture is made up to the 2-ml mark with dist. H_2O. After heating in a bath of boiling water for 15 min., the mixture is cooled in ice and shaken with pentanol (5 ml). The alcohol layer is rapidly removed and photometric estimation of the grey-blue colour carried out at 570 mμ on a Beckman spectrophotometer. An almost linear calibration curve can be obtained for 0 to 60 μg of neuramine acid. G. W. CAMBRIDGE

2756. **Two methods for the determination of glycogen in liver.** J. van der Vies (*Biochem. J.*, 1954, **57** [3], 410–416).—Two simple methods for determination of glycogen in liver are described. In both, the glycogen is extracted with trichloroacetic acid, but this procedure is not recommended for determination of glycogen in muscle. In the first method the glycogen is determined approximately by an iodine reagent as described by van Wagtendonk *et al.* (*Brit. Abstr. C*, 1946, 287). The colour intensity of the glycogen - I complex is determined spectrophotometrically at 650 mμ, and the amount of glycogen is ascertained from a calibration graph prepared from glycogen from the same origin as the sample. This gives an approximate value for the glycogen content, but the method is very convenient for serial determinations. The second method gives more accurate results. In this the liver extract is treated with alkali to remove interfering glucose; the glycogen is then hydrolysed to glucose by heating with aq. HCl, and the glucose is determined by the anthrone method of Morris (*Brit. Abstr. C*, 1948, 61).
J. N. ASHLEY

2757. **Method for determination of total lipids and water in brain tissue.** W. M. Sperry (*J. Biol. Chem.*, 1954, **209** [1], 377–386).—A semi-micro gravimetric method for determination of lipids in brain tissue is described; it avoids the errors which may arise from non-representative sampling, evaporation of water during disintegration of tissue, incomplete extraction, presence of non-lipid contaminants and oxidative degradation. Water is determined in the same sample (≃ 500 mg of homogeneous semi-liquid brain tissue) from which the lipids are extracted with chloroform - methanol (2 + 1). An apparatus is described for disintegration of tissue in a closed space without addition of a liquid or any other substance. J. N. ASHLEY

2758. **Venoarterial lipid, protein and polysaccharide differences.** N. Törnblom (*Acta Med. Scand.*, 1954, **149** [5], 369–375).—It has been shown that if the precipitate remaining after extraction of lipids by Bloor's method (*J. Biol. Chem.*, 1914, **17**, 377) is subjected to acid hydrolysis and again extracted by Bloor's method, an appreciable amount of substance is obtained having the same solubility properties as lipids. Hence, Bloor's method is not strictly quantitative, and the implications of this in the estimation of arterial-venous lipid differences are discussed. G. W. CAMBRIDGE

2759. **The quantitative assay of angiotonin.** J. Dekanski (*Brit. J. Pharmacol.*, 1954, **9** [2], 187–191).—A sensitive and relatively specific method is described for the quantitative assay of angiotonin (hypertensin) based on the pressor response obtained on injecting the material into the Dibenamine-treated rat blood pressure preparation. The preparation is about 20 times as sensitive as the cat preparation and 80 times as sensitive as the dog preparation. In the assay of plasma, interference by the presence of small amounts of pressor organic bases is eliminated by the use of Dibenamine, while vasopressin can be inactivated by treatment with sodium thioglycollate. A (2 + 2) design is used for the assays with doses in the range of 0·02 to 0·6 cat units. Doses may be repeated at 3 to 5-minute intervals without tachyphylaxis developing. The slope b was 56·1 mm of Hg and the index of precision, λ, was 0·036. Pressor and oxytocic effects of angiotonin appear to be similar to those of posterior lobe pituitary extract, and comparison of a 10 cat unit per ml solution of angiotonin with the pituitary standard showed it to contain pressor activity equivalent to 101·8 μg of pituitary per ml with fiducial limits of ± 14 per cent. On the isolated uterus of the rat, the solution of angiotonin contained oxytocic activity equivalent to 7·5 μg of pituitary per ml with fiducial limits ± 11 per cent. G. F. SOMERS

2760. **Colorimetric determination of carbamylamino acids and related compounds.** S. B. Koritz and P. P. Cohen (*J. Biol. Chem.*, 1954, **209** [1], 145–150).—The carbamylamino compound is condensed with diacetylmonoxime in presence of diphenylamine-*p*-sulphonic acid, and the resulting compound is determined colorimetrically. *Procedure*—Add 50 per cent v/v H_2SO_4 (6 ml), 1 per cent. aq. Na diphenylamine-*p*-sulphonate (0·1 ml), and 3 per cent. aq. diacetylmonoxime (0·25 ml) to the sample (3 ml). Mix the contents, cap the tubes and place them in boiling water for 10 min. Cool in cold water, and add 1 per cent. aq. $K_2S_2O_8$ (0·25 ml). Rapidly mix, re-cap, heat in boiling water for 1 min., cool immediately, and then protect from direct sunlight. After 10 min. determine the optical density at 550 mμ and ascertain the amount of carbamylamino acid from a standard graph. The reproducibility is ± 3 or ± 6 per cent. for amounts of 0·5 μ*M* or < 0·5 μ*M*, respectively. Any substance that contains a

ureide grouping will generally give some colour. Hydantoin gives a positive reaction, whilst the usual concn. of adenosine triphosphate do not interfere. Most of the amino-acids, thiourea, and urethane give no colour. In presence of 1 μM of carbamyl-L-glutamic acid, 5 μM of histidine or ornithine increases the colour value 23 per cent. Cysteine, cystine and methionine inhibit the colour, but inhibition by cysteine is overcome by increasing the amount of $K_2S_2O_8$. J. N. ASHLEY

2761. **A method for the determination of hyaluronic acid in the human intervertebral disc.** C. McClure, H. C. Holland and B. Woodhall (*Science*, 1954, **119**, 189).—A quantitative method is described for the determination of hyaluronic acid in sections of fresh frozen human intervertebral disc. After treatment with hyaluronidase, the N-acetylglucosamine liberated is estimated colorimetrically on addition of glacial acetic acid and *p*-dimethylaminobenzaldehyde. G. M. LEWIS

2762. **The Beck cuprimetric titration of the actomyosins from warm blooded hearts.** P. Cottier (*Schweiz. med. Wochschr.*, 1954, **84** [2, *ii*], 61–63).—A method is described for the extraction of actomyosin from mammalian heart. Beating hearts were removed from rabbits and transferred to liquid air within 3 sec. Portions of the ventricle (2 g) were homogenised at 4° C for 3 min. with 20 ml of extraction solution (0·5 *N* KCl with 0·03 *N* $NaHCO_3$). After stirring for 2 hr. and filtering, the pH of the filtrate was adjusted to 6·35 with acetic acid, and 5 volumes of distilled water (pH adjusted to 6·35 with PO_4''') were added. Following centrifugation at 2400 r.p.m. and washing twice, the ppt. formed dissolved on addition of 3·4 g of KCl and water to 100 ml. On the following day pptn. by distilled water was followed by solution in KCl (1·7 g per 50 ml). The biuret values and the cuprimetric determination of Beck (*Brit. Abstr. C*, 1951, 387) are reported for extracts of heart under various physiological and pathological conditions. G. W. CAMBRIDGE

2763. **The determination of purines in nucleic acids: a method applicable to materials with low concentration of nucleic acid.** I. W. McDonald *Biochem. J.*, 1954, **57** [4], 566–568).—A method is described for determination of adenine and guanine. Chloride and purine-containing compounds other than nucleic acids are first removed from the material, which is then hydrolysed with *N* H_2SO_4. The two purines are pptd. as their silver deriv., and after removal of Ag, they are separated by paper chromatography. The purine spots are identified under u.v. light; they are then cut out, extracted with 0·1 *N* HCl and the optical densities are determined spectrophotometrically at 250 mμ (for guanine) and 262 mμ (for adenine).

J. N. ASHLEY

2764. **Mercuric chloride test for determination of gamma-globulin fractions in serum.** K.-B. Mira and M.-D. Dušanka (*Acta Med. Scand.*, 1954, **149** [3], 237–242).—The results obtained by a rapid specific method for the determination of γ-globulin in small quantities of blood, based upon the precipitation of this fraction by $HgCl_2$, are compared with those obtained by paper electrophoresis. Good agreement has been achieved. Serum (0·1 ml) is pipetted into 5 ml of mixed phosphate buffer at pH 7·2 [0·067 *M* KH_2PO_4 (28 ml), 0·067 *M* Na_2HPO_4 (72 ml)]. The γ-globulins are precipitated by adding 0·047 per cent. $HgCl_2$ (0·4 ml) and the mixture is shaken vigorously. A blank determination is made with 5·4 ml of buffer and 0·1 ml of serum. The turbidity is determined after 20 min. in a Pulfrich photometer (Filter S43, tube 10 mm). The results are expressed as $HgCl_2$ units $= \frac{\text{extinction.}}{0{\cdot}07}$.

G. W. CAMBRIDGE

2765. **The staining of serum proteins, lipids and carbohydrates separated by paper electrophoresis.** C. Wunderly and S. Piller (*Klin. Wochschr.*, 1954, **32** [17–18], 425–432).—Details are given of the staining of lipids by the Sudan black - Ciba blue technique and carbohydrates by the HIO_4 - sulphite - fuchsin technique after their separatin by paper electrophoresis. G. W. CAMBRIDGE

2766. **Fractionation of serum proteins by zone electrophoresis in glass powder.** C. J. Bradish and N. V. Smart (*Nature*, 1954, **174**, 272–273).—The glass powder is contained in a vertical Perspex trough (27 × 18 × 2 cm) around which water at 2° C is pumped. The top of the trough is open, and the bottom is covered by a strip of calico under which is a row of 27 Perspex nipples attached to J-shaped capillary tube outlets. These are clamped to a horizontal bar, adjustment of which allows the outlet flow rate to be varied. At the ends of the trough are the ion-permeable partitions and electrode chambers through which buffer solutions are pumped at $\simeq$ 1 litre per min. during operation. The protein solution is injected 3 cm below the glass powder surface by a motor-driven syringe. Fractionation is carried out with a continuous flow of buffer and protein solution, or by a more rapid "no-flow" method. Clean separations of bovine albumin and γ-globulin are attained, but there may be slight contamination from the α- and β-globulins. C. E. SEARLE

2767. **The determination of serum protein fractions on filter-paper electropherograms by the biuret reaction, and some observations on the serum proteins of the oestrogenised immature pullet.** W. P. McKinley, W. A. Maw, W. F. Oliver and R. H. Common (*Canad. J. Biochem. Physiol.*, 1954, **32** [3], 189–199).—Horizontal-paper electrophoresis is carried out in a cold room (6° to 7° C) on Whatman 3 MM filter-paper 8·5 in. wide and 16 in. long at a potential of 200 V and a current of 9 to 10 mA. At the end of the run, usually 24 hr. or longer, a strip 1 in. wide is cut from either side, the remainder being replaced in the electrophoresis cabinet, but without the ends dipping into the electrode compartments. The strips are dried, and stained with naphthalene black in methanol containing 10 per cent. acetic acid. The protein pattern obtained is used as a guide for cutting the unstained undried paper into strips containing single protein fractions. The cut strip is enfolded in a piece of filter-paper about 1·5 in. wide, one end of which serves as a wick and dips into 1 per cent. saline, while the other end containing the strip from the electropherogram hangs in a test-tube receiver marked at 5 ml. A period of 2 hr. completes the elution. To each eluate, made up to 5 ml, if necessary, is added Weichselbaum's biuret reagent (5 ml), the contents are mixed and placed in a water-bath at 32° $\pm$ 0·5° C for exactly 30 min. A blank is prepared by eluting a portion of the filter-paper treated in the same way as the parts containing the protein. The optical densities are read on an Evelyn colorimeter with a 520 M filter. Standard curves of optical density plotted against protein (N × 6·25) are prepared from pooled sera.

The relative mobilities of the serum protein fractions of the domestic fowl, normal and oestrogenised, and of man are compared by use of aqueous and methanolic barbitone buffer.

E. C. BUTTERWORTH

2768. **The inter-action of dyes with proteins on paper with special reference to paper electrophoresis.** G. T. Franglen and N. H. Martin (*Biochem. J.*, 1954, **57** [4], 626–630).—The spectral characteristics of bromophenol blue, bromocresol green, azocarmine B, and naphthalene black 12 B are examined. Paper chromatography of azocarmine B shows that the dye is heterogeneous. Bromocresol green is the best dye to use for studies with proteins. By use of human albumin and human γ-globulin, it is shown that the plot of the dye bound against concn. of protein is not linear, and the characteristics of the relation of dye binding to protein concn. differ from one protein to another. Moreover, if two protein solutions of known concn. are mixed in known proportions, and the proteins are then separated by paper electrophoresis, the proportions of the two components calculated from dye binding techniques do not agree with the pre-determined ratios. Examination of a series of known mixtures shows that there is no consistent relationship from which a correction factor can be determined. Similar remarks probably apply to systems of four or more components. Electrophoresis of proteins on paper has considerable use as a qual. tool, but quant. analyses produced by existing staining techniques are of little value, and existing methods of analysis by paper strip electrophoresis require re-assessment.

J. N. ASHLEY

2769. **The micro-determination of cysteine in proteins.** C. Ghiglione and M. Bozzi-Tichadou (*Bull. Soc. Chim. Biol.*, 1954, **36** [4–5], 659–666).—Pure solutions containing 0·3 to 0·5 mg of cysteine are treated with $CdSO_4$ and NaOH, evaporated nearly to dryness and heated on a sand-bath at 200° C for 20 min. The residue is dissolved in H_2O, and 2·5 *N* H_2SO_4 and 0·1 *N* iodine soln. are added. After leaving the soln. 20 min. in the dark, saturated sodium borate soln. is added. Titration of the excess of iodine with 0·01 *N* $Na_2S_2O_3$ then enables the cysteine present in the original solution to be determined; average loss is 1·8 per cent. When methionine is present, the washed ppt. containing cysteine only is treated with oxalic acid, which precipitates Cd oxalate, leaving cysteine in solution to be determined as before. Proteins are hydrolysed and cystine is reduced to cysteine by refluxing with 5 *N* HCl and 90 per cent. formic acid. The acids are removed by adding water and evaporating to dryness several times under reduced pressure; the residue is dissolved in H_2O and adjusted to pH 6. Cysteine is then determined as in the presence of methionine. Cysteine contents of a number of proteins are recorded.

C. E. SEARLE

2770. **Polarographic determination of amino-acids.** D. R. Norton and N. H. Furman (*Anal. Chem.*, 1954, **26** [7], 1116–1119).—The reaction between amino-acids and phthalaldehyde is used to determine small amounts of glycine, alanine, tryptophan, aspartic acid, lysine and histidine. In a borate or carbonate buffer adjusted to pH 10·5, the reaction between amino-acids and phthalaldehyde is complete in approx. 2 hr. at reactant concn. of 10^{-3} *M*. Under these conditions the suppression of the reduction current of the second phthalaldehyde wave is directly proportional to the concn. of the amino-acid. As the reaction involves the amino group, the individual amino-acids, before their determination, must be separated from each other and also from NH_3, gelatin and other compounds that are known to react with the aldehyde. The accuracy of the method is comparable with other polarographic determinations. The reaction between phthalaldehyde and gelatin is discussed, and a linear relationship is observed between the aldehyde consumption and the gelatin concn.

D. BAILEY

2771. **A new method for the colorimetric estimation of amino-acids on paper chromatograms.** F. A. Isherwood and D. H. Cruikshank (*Nature*, 1954, **174**, 123–126).—2:4-Dinitrophenylamino-acids are separated from dinitrophenol (formed with excess of fluorodinitrobenzene) by dissolving in 91 per cent. H_2SO_4, in which they ionise as mono-acid bases, and extracting the dinitrophenol with benzene. After dilution of the H_2SO_4 to $\simeq$ 30 per cent., they are extracted with 2-methylbutan-2-ol - benzene (1 + 9). They are then extracted into saturated $NaHCO_3$ and estimated colorimetrically at 365 or 404·7 mμ against a standard of the same dinitrophenylamino-acid. Dinitrophenylglutamic acid is unstable in 91 per cent. H_2SO_4, so a modified procedure is described. Bright daylight causes destruction of the dinitrophenylamino-acids, but extreme precautions are not required. The accuracy for $\simeq$ 6-μg quantities is $\pm$ 5 per cent.

The initial separation of the amino-acids is by chromatography on filter-paper previously washed with 2 *N* acetic acid, H_2O and 10 *N* aq. NH_3; *n*-propanol - H_2O (4 + 1 by vol.) solvent is used for mono-amino mono-carboxylic acids and *n*-propionic acid - 2-methylpropan-2-ol - H_2O (6:3:1 by vol.) for the dicarboxylic acids. Excess of solvent is completely removed by steaming even when long drying at 100° C fails.

C. E. SEARLE

2772. **A paper-chromatographic method for the quantitative estimation of amino-acids.** A. L. Levy (*Nature*, 1954, **174**, 126–127).—Amino-acid mixtures are treated with 1-fluoro-2:4-dinitrobenzene at pH 9 (80 min. at 40° C), excess of the reagent being extracted with ether. The solution is acidified and re-extracted with ether to remove the dinitrophenyl derivatives of all amino-acids except arginine and histidine. Aliquots of the aq. and ethereal solutions are placed on adjacent corners of a filter-paper square, and chromatographed in toluene - $CHCl_3$ - pyridine - 0·8 *N* aq. NH_3 (5:3:1·5:3). After drying, the dinitrophenylarginine and α-dinitrophenylhistidine spots are cut out, and the remaining acids are chromatographed again at right angles in 1·5 *M* aq. phosphate buffer. The resulting spots are cut out together with blanks, and eluted with H_2O (15 min. at 55° to 60° C), cooled and estimated colorimetrically at 360 mμ (or 385 mμ for dinitrophenylproline). The method is applicable to hydrolysates from 0·2 mg of protein, and should be useful for analysing free amino-acids in the presence of NH_3, salts or proteins.

C. E. SEARLE

2773. **Separation of amino-acids by ascending unidimensional chromatography, and their determination by direct photometry of the chromatograms.** J. de Wael and R. Diaz Cadavieco (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [5], 333–346).—The paper-strip chromatograms of amino-acids in protein hydrolysates are obtained separately in 40 to 60 hr. by ascending unidimensional procedures and solvents such as (*i*) 0·067 *M* aq. phenol mixed with

phosphate buffer (pH 12), for aspartic and glutamic acids, serine, glycine, threonine and alanine, (*ii*) 0·067 *M* cresol mixed with KCl - NaOH - H_3BO_3 buffer (pH 8·4), for alanine plus arginine, tyrosine, histidine, valine and methionine, and (*iii*) a (1 + 1) mixture (0·067 *M*) of benzyl alcohol and butanol saturated with the pH 8·4 buffer, for proline, leucine and *iso*leucine. The solvents are subsequently removed by exposure for 2 hr. in a strong current of air at ≃ 25° C, followed by two successive washings with anhydrous ether. The colours are revealed by immersing the strips in a 0·2 per cent. soln. of ninhydrin (0·2 per cent. isatin for proline) in acetone containing ≃ 4 per cent. of acetic acid, followed by drying for 15 min. at 80° C in an atmosphere having a R.H. of 100 per cent. to ensure max. and reproducible values of colour density (*d*). The latter is determined for each of the bands by direct photometry by means of a slit of constant dimensions and a 518-mμ filter (589 mμ for proline). The concn. of the respective amino-acids are calculated from the curve relating *d* with distance along the strip. Variables affecting reproducibility and accuracy of the method are discussed, R_F values for the 15 amino-acids are listed, and quant. data for proteins extracted from egg and horse-serum albumins are given to show that the values compare well with those quoted in the literature. The mean error in determining *d* is ≃ 4 per cent. (*cf. Brit. Abstr. C*, 1949, 292).

W. J. Baker

2774. **A simple technique for extracting free amino-acids from biological fluids and extracts before their chromatographic separation.** S. Lissitzky, G. Césaire and R. Massonet (*Bull. Soc. Chim. Biol.*, 1954, **36** [4–5], 655–657).—Serum or plasma (1 ml) is absorbed on a piece of filter-paper (12 × 5 cm), which is dried in a current of air, cut into 16 pieces and extracted with 25 ml of acetone containing 1 per cent. v/v of conc. HCl. The mixture is centrifuged to remove fragments of filter-paper and the acetone is removed under reduced pressure at < 60° C. The residue is extracted with 3 ml of chloroform to remove some lipids and pigments, the chloroform being washed each time with dil. HCl, which is returned to the flask. The remaining solution of amino-acids is evaporated *in vacuo* at < 60° C, and the residue is taken up in water and chromatographed.

C. E. Searle

2775. **Microbiological determination of amino-acids.** H. Sarlet (*Ind. Chim. Belge*, 1954, **19** [5], 494–506).—A detailed description is given of the technical method developed by Dunn, Camien *et al.* of determining amino-acids. The method makes it possible to determine DL-alanine, L-arginine, L-, D- and DL-aspartic acids, glycine, L-histidine, L-*iso*leucine, L- and DL-methionines, L-phenylalanine, L-proline, L-serine, L-threonine, L-tyrosine, and L-valine.

H. Wren

2776. **A technique for electrophoresis combined with paper chromatography, "chromato-ionophoresis," applied to amino-acids and bases.** J. Blass, O. Lecomte and J. Polonovski (*Bull. Soc. Chim. Biol.*, 1954, **36** [4–5], 627–640).—Mixtures of amino-acids and bases are applied 3 cm from the centre of one side of a filter-paper square, and chromatography is carried out with butanol - acetic acid. The dried paper is cut down, and electrophoresis is carried out at right-angles to the direction of solvent flow, by means of a barbitone buffer, pH 8·6, and a p.d. of 10 V per cm (current 0·5 mA per cm) for 3½ hr. After drying, the substances are detected by means of ninhydrin (in butanol containing acetic acid and collidine). The serine and glycine spots are very close, but the method gives clearer separations of some substances than does one- or two-dimensional chromatography. The method is applied to the analysis of protein hydrolysates and the free amino-acids of normal and pathological sera.

C. E. Searle

2777. **Chemical estimation of progesterone in organic substrates.** J. Zander and H. Simmer (*Klin. Wochschr.*, 1954, **32** [23–24], 529–540).—A critical survey of progesterone estimation is given under the following headings: (*i*) extraction from blood, a modification of Butt's method; (*ii*) paper chromatographic separation of progesterone by means of 70 or 80 per cent. methanol as the stationary phase and light petroleum, ligroin or *n*-hexane as the mobile phase; (*iii*) localisation on paper by u.v. contact photography; (*iv*) elution and subsequent estimation by u.v. spectrophotometry at 200 to 270 mμ; and (*v*) detailed identification of the end product.

G. W. Cambridge

2778. **The determination of the amylolytic activity of saccharifying substances, and in particular, of fungal diastatic enzymes.** B. Drews and H. Specht (*Brauerei, Wissenschaft. Beil.*, 1954, **7** [7], 78–81).—The method for the determination of α-amylase activity, proposed by Sanstedt, Kneen and Blish (modified by Olsen and co-workers, *Cereal Chem.*, 1944, **21**, 533 and 1947, **24**, 299) is found to be affected by β-amylase. The method has been used to compare malt enzymes and fungal amylolytic enzymes. It was found that whereas the diastatic activity (Windisch - Kolbach) and α-amylase activity of fungal enzymes show parallelism, the corresponding values for malt enzymes do not. The α-amylase activity of fungal enzymes was as much as 20 times higher than that of some malts. With high α-amylase fungal enzymes the production of dextrins was no longer proportionate to the units of α-amylase activity as determined by the method of Sandstedt, Kneen and Blish.

G. B. Thackray

2779. **Liver catalase. I. A manometric determination of catalase activity.** R. E. Greenfield and V. E. Price (*J. Biol. Chem.*, 1954, **209** [1], 355–361).—A manometric assay of catalase activity is described; it utilises a pressure transducer and a continuous recorder system to record the pressure change of the O evolved from the H_2O_2 during the initial period of the reaction. The method differs from others in that stirring is more rapid and the manometrically measured O is compared with the substrate remaining (determined by titration with $KMnO_4$) after very short reaction times. The first-order reaction constant is proportional to enzyme concn. over a four-fold range of enzyme concn., but is independent of substrate concn. for 0·02 to 0·1*M* H_2O_2.

J. N. Ashley

2780. **The behaviour of blood lipase under the influence of changing altitude.** O. Wünsche (*Klin. Wochschr.*, 1954, **32** [25–26], 584–587).—Serum lipase determinations have been carried out on rabbits subjected to reduced barometric pressure. Blood serum (0·2 ml) is mixed with 4 per cent. Na citrate soln. (5 ml) buffered to pH 8·0 with 0·1 *N* NH_3 - NH_4Cl soln. After heating for 20 min. in a water-bath at 40° C, tributyrin (0·2 ml) is added and the mixture is shaken vigorously. The mixture is incubated for 3 hr. at 37° C and, after cooling,

0·12 per cent. alcoholic phenolphthalein soln. is added (2 ml) and the mixture is titrated to a faint pink end-point with 0·02 *N* NaOH. Lipase values are expressed in ml of 0·02 *N* NaOH after allowing for blank correction. G. W. CAMBRIDGE

2781. **Determination of the esterase activity in tissue suspensions from rabbit liver and kidney in experimentally induced renal disorders accompanied by hyperlipaemia.** A. Svanborg (*Acta Med. Scand.*, 1954, **149** [5], 349–353).—To avoid the difficulty of obtaining uniform aqueous emulsions of fatty acids, water-soluble esters have been used as substrates in lipase determinations. Tween 20 and Tween 40 were found to be suitable, and determinations of lipase activity in rabbit kidney and liver are reported. Approx. 0·5 g of tissue is homogenised in 0·9 per cent. NaCl (10 ml) at 4° C. The tissue suspension is added to 0·5 per cent. barbitone sodium (20 ml) together with Tween (0·5 g). After incubation at 37° C for 1 hr., the enzyme activity is arrested by the addition of a (9 + 1) mixture of ethanol and ether (100 ml). A control is carried out without the tissue suspension, which is added after ethanol - ether mixture. Both solutions are titrated to a pink colour with 0·05 *N* KOH with phenolphthalein as indicator. The results are expressed as millilitres of 0·05 *N* KOH per g dry weight of tissue.
G. W. CAMBRIDGE

2782. **Direct and continuous spectrophotometric assay of phosphomonesterases.** B. H. J. Hofstee (*Arch. Biochem. Biophys.*, 1954, **51** [1], 139–146).—A description is given of a method based on the liberation of salicylic acid from phosphorylmonosalicylic acid (substrate) by phosphomonesterases. The amount of salicylic acid liberated is determined directly on the reaction mixture by the increase in absorption at 310 mμ; at this wavelength the substrate has low absorption. The phosphatase activity can be determined over the pH range 2 to 11 on crude extracts corresponding to 1 mg or more of wet tissue. The preparation and properties of the substrate are detailed.
W. H. C. SHAW

2783. **Colorimetric determination of phenol oxidase.** S. Braesch (*Bull. Soc. Chim. Biol.*, 1954, **36** [4–5], 711–713).—Phenol oxidase is determined colorimetrically at 557·5 mμ by means of the red colour produced when the enzyme reacts with catechol in the presence of *p*-aminobenzoic acid. The solution is buffered at pH 9 (borate - boric acid) and the colour develops in 30 to 45 min. at room temp. The presence of alcohol accelerates colour formation and stabilises the colour. With potato enzyme, a direct relationship is obtained between concn. and scale reading for 1 to 7 units of enzyme. C. E. SEARLE

2784. **Creatine phosphokinase: assay and application for the micro-determination of the adenine nucleotides.** J. B. Chappell and S. V. Perry (*Biochem. J.*, 1954, **57** [3], 421–427).—A method for the assay of creatine phosphokinase is described. It involves determination of creatine by the method of Eggleton *et al.* (*Brit. Abstr. C*, 1943, 29) under conditions in which creatine phosphate is stable. The incubation mixture contains 0·05 *M* phosphate or aminotri(hydroxymethyl)methane buffer (pH 7·4), 0·005 *M* $MgCl_2$ or $MgSO_4$, 0·002 to 0·005 *M* adenosine diphosphate, 0·01 *M* cysteine (just neutralised before use), 0·005 *M* creatine phosphate, water and the enzyme solution. Incubation is effected at 30° C. The enzyme solution is diluted appropriately just before assay, and added to the incubation tube, which is equilibrated at 30° C for 4 min. The reaction is started by addition of the creatine phosphate, and is stopped after 3 to 5 min. by adding 0·05 *M* phenylmercury acetate in 50 per cent. aq. dioxan. The creatine liberated is determined for several concn. of enzyme. The results are plotted and the activity of the preparation is calculated from the slope of the graph. This method coupled with the creatine phosphokinase - hexokinase system provides an extremely sensitive means for determination of 0·5 to 5·0 μg of adenosine diphosphate and triphosphate. After addition of myokinase, the creatine phosphokinase - hexokinase system can be used for determination of small amounts of adenylic acid.
J. N. ASHLEY

2785. **The activity of the serum aldolase in patients with liver disease. A new enzymatic test.** F. Bruns and W. Puls (*Klin. Wochschr.*, 1954, **32** [27–28], 656–659).—The use of serum aldolase estimations as an index of liver function is described. The estimation is carried out as follows. Serum (1 ml) is treated with collidine buffer pH 7·4 (1 ml), 0·56 *M* hydrazine sulphate soln. (0·25 ml), 0·002 *M* iodoacetate (0·25 ml) and distilled H_2O (0·25 ml), followed by 0·06 *M* fructose diphosphate (0·25 ml). After heating for 1 hr. at 37° C, proteins are precipitated by 10 per cent. trichloroacetic acid (3 ml) and filtered off. One ml of filtrate is treated with 0·75 *N* NaOH (1 ml) for 15 min. at room temp., and, after the addition of 2:4-dinitrophenylhydrazine (1 ml, containing 1 mg in 2 *N* HCl) is heated for 10 min. at 38° C. The mixture is made up to 10 ml with 0·75 *N* NaOH, and, after 10 min., the intensity of the red colour is determined in a step-photometer (thickness 10 mm, Filter S53). Blank determinations are carried out and aldolase activity is expressed as the amount of fructose diphosphate (in cu. mm) that is broken down by 1 ml of serum in 1 hr. at 37° C under standard conditions (1 micro-mole of fructose diphosphate = 340 μg ≡ 22·4 cu mm). G. W. CAMBRIDGE

See also Abstracts 2788, 2828.

Drugs

2786. **Comparison of a spectrophotometric and the silver salt titration methods for the determination of theophylline in various pharmaceuticals.** J. P. Comer and W. W. Hilty (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [5], 287–290).—The spectrophotometric determination of theophylline in powders, injections, capsules, tablets and suppositories is based on the absorption of theophylline in 0·1 *N* HCl solution at 270 mμ. Results obtained by this method compared with corresponding results obtained by the silver salt titration method show that precision of the spectrophotometric method is as good and often better.
N. M. WALLER

2787. **The estimation of the component cardiac glycosides in digitalis plant samples. II. The estimation of the desgluco-glycosides and some observations on the production of ultra-violet fluorescence with trichloroacetic acid.** H. Silberman and R. H. Thorp (*J. Pharm. Pharmacol.*, 1954, **6** [8], 546–551).—A paper-chromatographic method is described for the reliable and reproducible separation and estimation of digoxin (**I**), digitoxin (**II**) and gitoxin (**III**) in the presence of original lanatosides and other glycosides present in crude

plant extracts. **II** can be separated (R_F value 0·7) in 2½ to 4 hr. by the ascending method by use of a mixture of $CHCl_3$, benzene, ethyl acetate and water (60:20:20:50 by vol.) as solvent; R_F values for **I** and **III** are 0·25 and 0·15, respectively. By using the descending method and extending the time to 18 to 22 hr., **I** and **III** can be separated by means of the same mixed solvent; slightly modified ratios of solvents are used for slightly longer or shorter development periods. The amounts in the spots should be in the range 0·5 to 3·0 μg and the temp. 22° to 24° C. After development, the chromatograms are sprayed with a 25 per cent. solution of trichloroacetic acid in $CHCl_3$. The effect of "ageing" in this reagent on the ultra-violet fluorescence given with various substances in the plant extract is reported. S. C. JOLLY

2788. **^{131}I-marked insulin. II. (Application and limitations of the detection method.)** E. Kallee (*Klin. Wochschr.*, 1954, **32** [21–22], 508–509).—Insulin marked with ^{131}I can be separated by paper electrophoresis at 100 V and 1 mA for 13 hr. at room temp. in barbitone buffer at pH 8·6. Autoradiography of the strips permits the detection of 10^{-9} g of marked insulin. The applications of this technique to the analysis of serum are discussed. G. W. CAMBRIDGE

2789. **An isotope-dilution assay for total penicillins.** M. Gordon, A. J. Virgona and P. Numerof (*Anal. Chem.*, 1954, **26** [7], 1208–1210).—An isotope-dilution assay for total penicillins in broth is described in which inexpensive ^{35}S-labelled penicillin is used. No degradation of penicillin is required and quantitative isolation of penicillin from broth (as in the gravimetric method) is unnecessary. Labelled penicillin is added to the broth, which is then adjusted to pH 2 to 2·5 with H_3PO_4 and extracted at 0° C with pentyl acetate. The pentyl acetate layer is extracted with cold phosphate buffer (pH 7) and the extract is again adjusted to pH 2 to 2·5 and extracted with $CHCl_3$. Penicillin is obtained from the $CHCl_3$ extract as its N-ethylpiperidine salt, which is recrystallised, and its activity is determined. Penicilloic acid, the most likely contaminant of penicillin in broth, does not interfere. The 95 per cent. confidence limits are ± 5·9 per cent. on the mean of duplicate assays. Samples containing a total of 10,000 units in a convenient volume can be assayed by this procedure. D. BAILEY

2790. **Separation of tetracycline, chlortetracycline and oxytetracycline by paper chromatography.** H. L. Bird, jun., and C. T. Pugh (*Antibiot. & Chemother.*, 1954, **4** [7], 750–752).—The method is applied to the pure compounds, to fermentation liquors and, in conjunction with other solvent systems for preliminary classification, to the identification of the three antibiotics in cultures from soil.

Procedure—Cut Whatman No. 1 filter-paper along the short length of the sheet into 19 × 46·5-cm strips. Impregnate with buffer, pH 3·0 (60 g of KOH, 70 ml of H_3PO_4 and water to 1 litre) and dry in air. In a line 7·5 cm from one end of the strip place four spots (⩾ 1 cm in diameter) each containing 0·2 to 0·5 μg of the antibiotics. Chromatograph in a closed container using the descending technique with water-saturated ethyl acetate for 20 hr. at 23° C, allowing the solvent to drip off the end of the strip during development. After development, allow most of the solvent to evaporate, hang the strips for 5 to 10 sec. in the vapour of strong aq. NH_3 and then dry them in air. The spots are located on the strips by a bio-autographic technique similar to that of Goodall and Levi (*Nature*, 1946, **158**, 675). W. H. C. SHAW

2791. **Enzymatic microbiological analysis of chloramphenicol esters.** C. Trolle-Lassen (*Arch. Pharm. Chemi*, 1954, **61** [12], 435–452).—A weighed sample of a chloramphenicol ester is dissolved in the min. quantity of ethanol and diluted with a buffer soln., pH 6, to give a suspension containing approx. 0·1 mg of ester per ml; 1 mg of lipase B per ml is then added. The mixture is incubated at 37° C for 4 hr. and then compared by a microbiological method with a chloramphenicol standard. Examination of the significance of temp., pH, reaction time and ratio of enzyme to ester (chloramphenicol stearate) led to the prescribed conditions under which hydrolysis is nearly complete. N. M. WALLER

2792. **Spectrophotometric determination of acetylsalicylic and salicylic acids.** R. B. Tinker and A. J. McBay (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [5], 315–317).—Acetylsalicylic acid and salicylic acid are determined in capsules and tablets by a method based on their absorption in a $CHCl_3$ soln. at wavelengths of 308 mμ and 278 mμ. Equations are presented for the calculation of the percentage of each constituent. The method is accurate to at least ± 0·2 per cent. for each. N. M. WALLER

2793. **Potentiometric method of assay for sodium *p*-aminosalicylate.** J. R. Stockton and R. Zuckerman (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [5], 273–275).—An aqueous soln. of the sodium salt of *p*-aminosalicylic acid is diluted with a mixture of ethylene glycol and *iso*propanol and titrated potentiometrically with 0·4 to 1·0 *N* $HClO_4$ in these solvents. The method determines the extent of decomposition of the Na salt, and is applicable to tablets, granules and injectable preparations, and to mixtures with streptomycin. N. M. WALLER

2794. **Rapid colorimetric method for determination of *iso*nicotinic acid hydrazide [isoniazid] in blood plasma.** B. Prescott, G. Kauffmann and W. D. James (*Proc. Soc. Exp. Biol. Med.*, 1953, **84** [3], 704–706).—The method described is based on the condensation of isoniazid with glutaconic aldehyde formed by alkaline hydrolysis of 4-pyridylpyridinium dichloride. Two ml of isoniazid solution containing 3 per cent. w/v of aq. trichloroacetic acid are treated in succession with 0·5 ml of 1 per cent. aq. 4-pyridylpyridine, 0·7 ml of 2 *N* NaOH and 0·5 ml of 2 *N* HCl. The mixture is set aside for 15 min. after making to 5 ml and the intensity of the colour is measured at 425 mμ. The intensity of the yellow colour developed varies logarithmically with the concentration of isoniazid and conforms to the Beer - Lambert law. Blood plasma levels in mice and guinea pigs after oral administration of 5 and 50 mg, respectively, of isoniazid, are found to reach a maximum within 30 min. to 2 hr. and to fall to low levels within 18 hr. I. JONES

2795. **Analytical total acid hydrolysis of dextrans.** R. J. Dimler, H. A. Davis, G. J. Gill and C. E. Rist (*Anal. Chem.*, 1954, **26** [7], 1142–1146).—An investigation to determine the optimum conditions for complete acid hydrolysis of dextran that may be used either in determining the concn. of dextran

solutions or in liberating the constituent monosaccharide units for identification is described. The same max. yield of reducing sugars (96 to 98 per cent.) and concurrent loss of D-glucose are obtained in 50 to 80 min. with 4 *N* H_2SO_4 and 2 *N* HCl at 100° C and *N* H_2SO_4 at 120° C, and in 150 to 180 min. with 2 *N* H_2SO_4 and *N* HCl at 100° C. A standardised procedure is described for analytical hydrolysis with a 0·5 per cent. solution of dextran in 4 *N* H_2SO_4 heated for 75 min. at 100° C. The observed reducing power is corrected for an approx. 4 per cent. loss of D-glucose during hydrolysis. The method is applicable to different types of dextran, but not to laevans, probably owing to extensive decomposition of the relatively acid-sensitive ketohexoses. D. BAILEY

2796. **Physico-chemical characterisation of clinical dextran.** J. A. Riddick, E. E. Toops, jun., R. L. Wieman and R. H. Cundiff (*Anal. Chem.*, 1954, **26** [7], 1149–1155).—The existing chemical and physical methods for the determination of clinical dextran are reviewed and investigated. The fractionation procedures for determining mol. wt. distribution are studied. A comparison made of wt. average mol. wt. determined by light scattering photometry and viscometry reveals that the former method is most satisfactory. Both fractional pptn. with methanol and light-scattering photometry are adapted for routine control. D. BAILEY

2797. **Determination of nicotine in tobaccos.** A. Ollero Gómez and R. Cota Galán (*An. Soc. Esp. Fís. Quím., B*, 1954, **50** [4], 413–420).—The nicotine is determined directly on aq. extracts of tobacco, without previous isolation, by colorimetric reaction with cyanogen bromide and 1-naphthylamine. About 30 to 60 mg of powdered tobacco and 0·2 g of MgO are shaken with 100 ml of H_2O for 1 to 2 hr., the extract is filtered, and to 5 ml of extract are added 5 ml of 0·2 per cent. 1-naphthylamine soln. (in 80 to 85 per cent. ethanol) and 1 ml of cyanogen bromide solution. After 3½ hr. in darkness, the colour is measured and the nicotine content is read off from graphs. Influence of pH and interfering substances are studied and comparisions are made with a gravimetric method.
L. A. O'NEILL

Food

2798. **The determination of lead in foodstuffs. Report of the Lead Panel of the Metallic Impurities in Foodstuffs Sub-Committee of the Analytical Methods Committee of the Society for Analytical Chemistry** (*Analyst*, 1954, **79**, 397–402).—A method for determination of Pb in foodstuffs is described in detail. Preliminary treatment of the sample is either by wet ashing with H_2SO_4 and HNO_3 and finally $HClO_4$ or by dry ashing with $Mg(NO_3)_2$ at ≯ 500° C, the residue in each procedure being dissolved in HCl. In the absence of significant amounts of PO_4''', Bi or Fe, section (*a*) of the method below may be omitted. (*a*) The soln. is made just alkaline to methyl red with aq. NH_3 and then acid with an excess (10 ml) of 5 *N* HCl. After addition of NaI, liberated I is removed with sodium metabisulphite and the liquid is extracted with a soln. of diethylammonium diethyldithiocarbamate in $CHCl_3$. Dil. H_2SO_4 is added, the solvent is removed and the residue is heated with $HClO_4$ and dissolved in HCl. (*b*) Sodium metabisulphite, ammonium citrate and bromothymol blue are added and aq. NH_3 until the indicator is a full blue colour, and, after addition of exactly 10 ml of $CHCl_3$, the soln. is treated with dithizone soln. until there is a slight excess. The separated $CHCl_3$ layer is washed with a KCN wash liquid (prep. described) to remove uncombined dithizone, and its optical density is measured at 520 mμ. The lead content is ascertained from a calibration graph prepared with a standard lead soln.
A. O. JONES

2799. **The aspecific detection of preservatives in foods by a simple fermentation test with special reference to cured meat products.** D. A. A. Mossel (*Analyst*, 1954, **79**, 443–446).—As an alternative to tedious chemical methods, a fermentation test can be applied as a non-specific means of detecting certain toxic antimicrobial agents (*e.g.*, derivatives of bromoacetic acid and phenylmercury compounds) sometimes added to non-sterile canned solid foods. With solid foods (particularly cured meat products), an extract with 0·5 per cent. tartaric acid soln. and a similar extract with 0·1 per cent. NaOH soln. are sterilised at 80° C at pH 3 and are then enriched with sterile yeast extract and dextrose. The pH is adjusted to 4 and the extracts are inoculated with sufficient yeast to provide 10^4 live cells per ml. After incubation in Einhorn tubes for 24 to 30 hr. at 24° ± 2° C, the vol. of gas evolved is measured and compared with controls. Bromoacetic acid derivatives can be detected at concn. equiv. to 5 mg of Br per kg, hexamethylenetetramine at 0·1 per cent., H_3BO_3 at 0·5 per cent. and benzoic acid at 0·1 per cent. NaCl, KNO_3 and $NaNO_2$ do not interfere at the concn. usually found in these products. The test cannot be used for detecting antibiotics such as penicillin, as these show very weak inhibition of yeast activity. A. O. JONES

2800. **Colorimetric determination of starch.** J. Carles (*Bull. Soc. Chim. Biol.*, 1954, **36** [4–5], 705–710).—A rapid and sensitive colorimetric method for the micro-determination of starch is described. The sample (< 50 mg) is crushed with 10 ml of $CaCl_2$ (2 g per ml) made just alkaline to phenolphthalein, 1 ml of 0·5 *N* acetic acid is added and the mixture is heated for 20 min. on a steam-bath. To 10 ml of the solution, suitably diluted, is added 1 drop of iodine reagent (1·5 g of KI and 1·2 g of I in 100 ml), and, after 15 min., the colour of the solution is measured. A standard graph is made for pure maize starch analysed titrimetrically and polarimetrically. This graph can be used for other starches by applying a conversion factor. Most reliable readings are attained in the scale region 0·1 to 0·7; 3 divisions are equiv. to 1 μg of starch. C. E. SEARLE

2801. **Quantitative analysis of sugars in beet molasses by partition chromatography on paper.** L. Cronenberger and L.-G. Cuzin (*Bull. Soc. Chim. Biol.*, 1954, **36** [4–5], 715–717).—De-proteinised and de-salted beet molasses are chromatographed on paper by means of butanol - acetic acid - H_2O (40:10:50) as solvent. The chromatogram is overrun for 2½ days. The sugars are located with aniline phosphate. Raffinose, saccharose, glucose and fructose are found in the molasses, as well as sugar that is probably melibiose. The proportions of the sugars vary widely in different molasses.
C. E. SEARLE

2802. **Design and operating technique of a vacuum drying oven. II. Solids in cane molasses.** S. D. Gardiner and F. J. Farmiloe (*Analyst*, 1954, **79**, 447–453).—With the suitable vacuum oven

previously described (*Anal. Abstr.*, 1954, **1**, 590), a drying method at 69° to 70° C was devised for cane molasses, values for true solids being determined to within ± 0·1 per cent. The sample is diluted with water according to the solid content indicated by the refractometer, and a calculated vol. of the soln. is incorporated with dry aluminium powder as surface extender in an aluminium dish. The dish is placed in the oven at 50° C and the temp. is gradually raised to 70° C, at which it remains for the drying period. Loss of wt. is determined at stipulated intervals in order that a graph showing percentage loss with time may be drawn; a method of correcting this graph for decomposition of the solids is also described. An ash correction is derived to permit solids to be determined by refractometry with the use of sucrose tables, which obviate the need for a long difficult determination by drying. Equations are derived relating true solids to refractometer solids, invert sugar and sulphate ash. An additional correction for Na_2SO_4 and K_2SO_4 involving the use of a flame photometer, gives still more accurate results. A. O. JONES

2803. **An examination of Scottish heather honey.** T. J. Mitchell, E. M. Donald and J. R. M. Kelso (*Analyst*, 1954, **79**, 435–442).—Ling honey from the nectar of *Calluna vulgaris* is unique among European honey in colour, taste, viscosity and the property of thixotropy. Forty-two samples (of which 30 were predominantly ling honey) from widely scattered districts in Scotland and Northumberland were examined to find a correlation between these properties and the content of N, mineral matter and colloid. The colloid was determined gravimetrically by pptn. of the strained honey with trichloroacetic acid, N by the Kjeldahl method and the mineral matter by charring in presence of a little H_2SO_4 and final ignition at 800° C, an arbitrary 10 per cent. being deducted to allow for replacement of other groups by SO_4''. The pH was determined by glass electrode with a 10 per cent. soln. and free acid by the standard A.O.A.C. procedure. Moisture content was ascertained from the refractive index by use of Chataway's tables (*Brit. Abstr. B*, 1932, 654) correlating moisture, viscosity and *n*. The thixotropy ratio was determined by timing the descent of a steel ball in the honey before and after stirring. Correlation was found between the pH and ash content and some evidence of relationship between colloid content and the total N and the thixotropy. Variation in the floral sources may account for a certain inexactness in the relation between these properties. A. O. JONES

2804. **The volatility of sodium chloride and the determination of salt and ash in bread.** J. Terrier (*Mitt. Lebensm. Hyg., Bern*, 1954, **45** [2], 111–115).—Sodium chloride is determined by moistening 2·5 g of the powdered bread with 1·5 ml of approx. *N* Na_2CO_3, drying and carbonising. The chloride is extracted with water and determined by the Volhard method. Details are given for obtaining a white ash of bread without loss of NaCl. No loss occurs on heating NaCl alone to 375° C, but if mixed with flour losses occur that may be prevented by adding Na_2CO_3. N. E.

2805. **Standardised procedures for the Babcock test for milk.** W. A. Cordes, J. E. Edmondson, T. I. Hedrick, E. O. Herreid, L. M. Lambert, J. J. Willingham and B. Heinemann (*J. Dairy Sci.*, 1954, **37** [6], 761–768).—Detailed instructions are given for sampling, for the preparation and preservation of samples, and for carrying out the test. W. H. C. SHAW

2806. **Methods of determining the percentage of total solids in milk by means of the lactometer.** B. Heinemann, J. Cosimini, E. L. Jack, J. J. Willingham and B. M. Zakariasen (*J. Dairy Sci.*, 1954, **37** [7], 869–876).—A report is given of a collaborative investigation into methods for estimating total milk solids by calculation from the lactometer reading and the percentage of fat determined by a modified Babcock test (*J. Dairy Sci.*, 1950, **33**, 685). Results on 78 whole-milk samples show that the modified Sharp and Hart equation (*J. Dairy Sci.*, 1946, **29**, 87) gives a satisfactory estimate (within the limits of error) of the total solids determined by the Mojonnier method. The standard error found for each part of the assay is given. Procedures for calibrating the lactometer and for carrying out the lactometer test are detailed. W. H. C. SHAW

2807. **Tetrazolium reduction test for quality control of milk.** H. Laxminarayana and K. K. Iya (*Indian J. Dairy Sci.*, 1954, **7** [2], 83–92).—The use of tetrazolium (2:3:5-triphenyltetrazolium bromide) as an oxidation - reduction indicator in the bacteriological testing of milk has been previously described (Laxminarayana and Iya, *Curr. Sci.*, 1952, **21**, 124). Reduction by bacteriological activity converts it irreversibly to a cherry-red compound. The reaction has been studied with milks of different grades and the results have been compared with those of the methylene blue and the resazurin tests. One ml of 0·1 per cent. tetrazolium soln. is added aseptically to 10 ml of milk in a sterile Pyrex-glass test-tube (6 in. × $\frac{5}{8}$ in.). The tubes are closed, inverted 2 or 3 times and incubated at 37° ± 0·5° C, in the absence of direct sunlight. The colours of the tubes are compared at frequent intervals with reference standards. The test compares well with the accepted tests for detecting poor quality milk. G. B. THACKRAY

2808. **Factors affecting the sensitivity and accuracy of the phosphatase test [for dairy products].** G. P. Sanders, O. S. Sager and J. A. Hupfer (*J. Dairy Sci.*, 1954, **37** [6], 698–710).—The effects of variation in pH, buffer concentration, incubation period and temp. on the relative sensitivities and linearity of results given by three modifications of the phosphatase test have been investigated, and optimal conditions have been found for each modification. Protein pptn., the use of 2:6-dibromoquinonechloroimine as colour reagent, and the effect of added phosphates have also been studied. The criteria for pasteurisation with each modification of the test are discussed. W. H. C. SHAW

2809. **The determination of alkaline phosphatase of milk and the estimation of the degree of heating.** O. Saingt and J. Jacquet (*Ann. Falsif.*, 1954, **47** [2], 91–103).—The substrate chosen is Na β-glycerophosphate. The following method is proposed. Measure 0·1 ml of milk into each of two test tubes. Heat one tube in a bath of boiling water for 5 min. Add to each, 5 ml of buffered substrate solution (aq. Na β-glycerophosphate, 1·5 g per litre, saturated with $CHCl_3$, to which is added 11·5 g per litre of Na barbitone), plug the tube with cotton wool and incubate it for 24 hr. at 37° C. Immerse both tubes in boiling water for 5 min. Add 10 ml of aq. trichloroacetic acid (10 per cent. w/v), and allow

to stand for 10 min. Filter, and transfer 0·1 ml of the filtrate to a clean test-tube and add 1 ml of sulphuric - molybdate reagent (50 g of ammonium molybdate in 1 litre of N H_2SO_4) and 2 ml of aq. quinol soln. (20 g per litre). Allow to stand for 10 min. Change the green colour produced to blue by adding alkaline Na_2SO_3 (500 ml of Na_2SO_3, 150 g per litre, added to 2 litres of Na_2CO_3, 200 g per litre). Measure the colour in a photo-electric colorimeter. Deduct the figure found for phosphorus in the blank determination. The results are expressed in units of phosphatase activity defined as follows: 1 unit corresponds to the liberation of 1 mg of phosphorus, as inorganic phosphorus, from a suitable specified substrate, the experiment being conducted at the optimum pH for that substrate. Results are given for crude and butter milks and for abnormal milks. The reproducibility is $\pm$ 0·1 unit per ml. G. B. THACKRAY

2810. **A wettability method for powdered milk.** U. S. Ashworth and H. Gunthardt (*J. Dairy Sci.*, 1954, **37** [7], 863–868).—A simple empirical wettability test is proposed for powdered whole milk. The relationship between wettability and ease of reconstitution is discussed. W. H. C. SHAW

2811. **Relationship between *in vitro* enzymatic digestibility and *in vivo* protein evaluation of powdered whey.** R. M. DeBaun and W. M. Connors (*J. Agric. Food Chem.*, 1954, **2** [10], 524–526).—The nutritional value of whey powder is studied in relation to the protein-sugar (Maillard) reaction. This reaction occurs during processing and storage and involves chiefly the sugar aldehyde group and lysine. In the method proposed, lysine is liberated by preliminary digestion with trypsin and then estimated microbiologically with *Strep. faecalis* (ATCC 9790). An approx. linear relationship is found between the results and the biological values obtained in rat-feeding experiments on roller- and spray-dried whey powders stored under different conditions. The method is used to predict the results of animal evaluation with fair precision and is also applicable to milk powders, milk concentrates and whey protein fractions.
W. H. C. SHAW

2812. **The colorimetric determination of ammonia nitrogen in cheese.** E. Bernhard (*Mitt. Lebensm. Hyg., Bern.*, 1954, **45** [2], 115–122).—A method for determining ammonia in cheese with Nessler reagent is described. Sodium citrate is added to increase the colour intensity, no heat is used and only free ammonia is estimated, as proteins, amino-acids, etc., do not react under the conditions of test. The instructions must be very closely followed or a fresh calibration curve is required for each test. Mix at 45° C, 10 g of cheese into a smooth paste with 0·5 N sodium citrate (40 ml). Add 50 ml of H_2O and stir the emulsion for half an hour at 45° to 50° C. Make up to 200 ml. To a 25-ml aliquot add dropwise 20 per cent. trichloro-acetic acid (25 ml), shake and filter. Neutralise 25 ml of the clear filtrate dropwise with 10 per cent. NaOH, make up to 100 ml, add 2 g of Permutit and shake the mixture vigorously for 3 minutes. Wash the Permutit with water, and transfer it to a 100-ml calibrated flask, add 10 per cent. NaOH (2 ml), water (68 ml) and Nessler reagent (2 ml), and make up to the mark with water. Measure the extinction after exactly 15 min. P. S. STROSS

2813. **Infra-red determination of diphenyl in citrus fruits.** W. F. Newhall, E. J. Elvin and L. R. Knodel (*Anal. Chem.*, 1954, **26** [7], 1234–1236).—A quant. method is described for the direct determination of diphenyl (absorbed from cartons) in orange oil obtained by steam distillation of minced peel or juice of citrus fruits by a continuous liquid - liquid extraction technique. The diphenyl content of the dried oil is determined by infra-red absorption over the range 13·90 to 14·60 μ at slit widths of 1·55 and 1·90 mm for 0·1 and 0·4-mm spacers, respectively. Variations in the optical properties of individual orange-oil samples do not affect the accuracy of the results (average recovery 98 per cent., standard deviation $\pm$ 4·24 per cent.), when calculated by a base-line technique. The accuracy is good for samples containing 0·08 to 0·8 per cent. of diphenyl. D. BAILEY

2814. **Spectrophotometric determination of theobromine and caffeine in cocoa powders.** D. T. Englis and J. W. Miles (*Anal. Chem.*, 1954, **26** [7], 1214–1218).—Cocoa powder mixed with MgO and Celite is thoroughly extracted with hot water and the extract is neutralised with 0·1 N H_2SO_4 to pH 6·5, treated with Zn acetate and $K_4Fe(CN)_6$ solutions and filtered. An aliquot of the filtrate is purified by adsorption on a fuller's earth - Celite column and elution with 0·1 N NaOH. The eluate is acidified with N HCl and the absorption is determined at 272·5 and 310 mμ. A calibration curve gives the theobromine content of the solution. Caffeine is obtained from the aq. extract of cocoa powder, after treating with $K_4Fe(CN)_6$ solution and filtering, by making alkaline with conc. aq. NH_3 and extracting with $CHCl_3$. The extract is washed with N H_2SO_4 and its absorption is determined at 276 and 310 mμ. A calibration curve gives the caffeine content of the solution. D. BAILEY

2815. **Colorimetric determination of trace metals in beer and in brewing materials. VIII. Determination of arsenic.** W. J. Stringer (*J. Inst. Brewing*, 1954, **60** [3], 249–255).—A method for the determination of arsenic is described. The method is a modification of the Gutzeit method and is suitable for routine examination of beer and brewing materials. After wet oxidation (see *Brit. Abstr. C*, 1952, 115), cool the residue and add 0·5 g of hydrazine sulphate, and heat strongly for 10 min. Dilute with 25 ml of distilled water and wash into the reaction vessel with a further 25 ml of distilled water. The vessel, which already contains 10 g of arsenic-free Zn, consists in a 100-ml Erlenmeyer flask fitted with an absorption tube containing 2 ml of aq. Pb acetate soln. (5 per cent.). Above this a Perspex attachment is cemented to the absorption vessel, inside which are two Perspex collars with mercuric bromide paper between them. A Perspex screw washer tightens the assembly. There are three pairs of collars that allow the formation of stains 15, 10, or 5 mm in diameter. After 1½ hr. the stain is treated with CdI_2 and compared with standards prepared on the same day. Full details are given, including a specification for the arsenic-free Zn to ensure a satisfactory rate of evolution, and a method of preparing the mercuric bromide papers. For samples containing much mineral matter a distillation procedure is given. The apparatus made in Pyrex glass or silica consists of a 70-ml distillation flask, into which a thermometer is fixed and which has a side arm for introducing N or CO_2, a small condenser and a receiver, which includes a trap. Wash the residue from the

oxidation, after treatment with $(NH_4)_2SO_4$ but before adding hydrazine sulphate, into the distillation flask with 20 ml of distilled water, add 2 ml of aq. KBr (30 per cent.), 10 ml of conc. H_2SO_4 and, after mixing, 10 ml of conc. HCl. Distil for 10 min. into 2 to 3 ml of conc. HNO_3, adding conc. HCl drop by drop and keeping the temperature at 120° C. Disconnect the receiver and continue the distillation to wash the joint. Wash the distillate into a Kjeldahl flask and add 8 ml of conc. H_2SO_4. Treat with $(NH_4)_2SO_4$ and hydrazine sulphate and complete as above. The method has proved reliable for 0 to 12 μg of As_2O_3.

G. B. THACKRAY

2816. **Determination of acetaldehyde by bisulphite methods and their application in the analysis of wines.** J. F. Casas Lucas (*Rev. Cienc. Apl.*, 1954, **8** [2], 103–111).—A critical review of direct and indirect bisulphite methods is given. Factors influencing the formation, stability and dissociation of the bisulphite compounds, and the iodimetric titration of SO_3'' are considered. An indirect method, which can be used in presence of substances that react with I in neutral and alkaline but not acid media, involves addition of aq. ethanol and aq. $NaHSO_3$ to the neutral aldehyde solution, followed by a citric acid buffer of pH 3, titration with iodine solution, and comparison with a blank determination. For the determination of acetaldehyde in wine, a direct method of determination in which the bisulphite compound is decomposed by addition of $NaHCO_3$, and the SO_3'' is titrated with iodine solution is most satisfactory. Before determination, the acetaldehyde must be distilled as the wine contains other carbonyl compounds. If this distillation is carried out at ≃ pH 9, the value obtained will represent the free acetaldehyde in the wine and the acetaldehyde present as a bisulphite compound.

L. A. O'NEILL

2817. **Conductimetry of wines.** I. Mareca Cortés (*Inf. Quim. Anal.*, 1954, **8** [3], 86–89, 85).—The values for the sulphate contents of various wines determined conductimetrically are within 1 per cent. of those obtained gravimetrically. A solution of 0·25 *N* Ba acetate from a micro burette marked at 0·1-ml intervals is used. The method of conductimetry need not be laid down rigidly, and neither special electrodes nor a set distance between them is used. The wine (10 ml) is diluted with 20 ml of distilled water for the test.

H. PRITCHARD

2818. **Rapid method for determination of oil in oil-seeds.** T. François and G. Bleicher (*Compt. Rend. Acad. Agric. France*, 1954, **40** [8], 314–316).—The sample (5 g) of soft seed (which may include soya-beans) is treated for three periods of 3 min. with three successive portions of 30 ml of light petroleum (boiling range 40° to 60° C) in a micro-mill, which consists in a centrifuge tube provided with a screw-down cover, in which is mounted a stainless-steel agitator revolving at 15,000 to 18,000 r.p.m. Under these conditions, all cellular tissue is disintegrated. The micellae (after settling for a short time) are successively decanted on to a sintered-glass filter, and the oil is determined gravimetrically in 25 ml of the mixed filtrate and washings, after these have been made up to 150 ml. Materials such as copra or palm-nuts should be comminuted (dry) in a Turmix-type mill before weighing-out and further treatment. The results obtained compare favourably as regards reproducibility with those obtained by the standard (Soxhlet extraction) method.

P. S. ARUP

2819. **The determination of low bromine absorption values.** V. W. Reid and J. D. Beddard (*Analyst*, 1954, **79**, 456–458).—The method described is applicable to bromine numbers < 1. The reagent is Br dissolved in glacial acetic acid adjusted to be equiv. to a 0·1 *N* $Na_2S_2O_3$ soln. A sample of suitable size is cooled in ice water for 10 min., 10 ml of Br soln. are added and the mixture is again cooled for 3 min. KI soln. is then added and the liberated I is titrated with 0·01 *N* $Na_2S_2O_3$ soln. A blank determination is made similarly without the sample. The method should find application where the properties of a product are adversely affected by traces of unsaturated compounds.

A. O. JONES

2820. **Determining the density of triglycerides and esters by the falling-drop method.** R. P. A. Sims (*J. Amer. Oil Chem. Soc.*, 1954, **31** [4], 144–147).—The determination of the densities of triglycerides and other esters by the falling-drop method is described. The densities are determined to within ± 0·0003, when the behaviour of two drops is compared, and to ± 0·0002, when only one drop is used. Triolein and methyl erucate are used as reference materials and the media are aq. solutions of ethanol and methyl cyanide. The influence of the medium and the effect of variation of temp. on the densities are discussed. The method is rapid, but less accurate than other micro methods.

D. BAILEY

2821. **Determination of the phosphorus content of lipids.** W. D. Harris and P. Popat (*J. Amer. Oil Chem. Soc.*, 1954, **31** [4], 124–127).—The molybdenum blue methods for the determination of P in organic compounds are reviewed and a modified method is described. The oil sample (0·07 to 0·10 g) is heated with 70 to 72 per cent. $HClO_4$ (1·0 ml) and conc. HNO_3 (1 drop) until the oxidation reaction subsides. Further conc. HNO_3 (2 drops) is added and the digestion is continued until fumes of $HClO_4$ appear. After cooling, the digest is diluted with a little H_2O, treated with 5 per cent. aq. ammonium molybdate (1 ml) and reducing solution [*p*-methylaminophenol sulphate (Metol) (0·5 g), $NaHSO_3$ (12·5 g) and Na_2SO_3 (2·4 g) in 100 ml] (2 ml) and diluted to 25 ml. The optical density of the sample is determined against a blank at 820 mμ and the P content is determined from a reference graph. Colour stability is good and the accuracy is within 1 per cent. for 1·0 to 5·0 p.p.m. of P.

D. BAILEY

2822. **Detection of adulteration of butter with vegetable oils by means of the tocopherol content.** J. H. Mahon and R. A. Chapman (*Anal. Chem.*, 1954, **26** [7], 1195–1198).—The tocopherol level of butter oil is low (0·002 to 0·005 per cent.), whilst that of most vegetable oils, with the exception of coconut oil, is considerably higher. The method described for detection of adulteration of butter with vegetable oils is based on the tocopherol content. The butter oil is dissolved in light petroleum and extracted with 60 per cent. H_2SO_4 to remove synthetic butter colours and vitamin-A alcohol. Carotene, which is not removed by this procedure, gives an "apparent tocopherol" value and is determined by the absorption of the extracted light petroleum and fat solution. The "total" tocopherol is determined on the extracted fat solution by the Emmerie and Engel colorimetric method, and a correction is applied for the carotene content. The application of the method to butter-fats adulterated with various animal and vegetable

fats and oils is discussed. Limits for tocopherol contents are suggested. D. BAILEY

2823. **A chromatographic method for the estimation of oleic and linoleic acids in the presence of straight-chain saturated fatty acids.** W. M. L. Crombie, R. Comber and S. G. Boatman (*Nature*, 1954, **174**, 181–182).—A reversed-phase chromatographic procedure is proposed for estimating oleic and linoleic acids in the presence of straight-chain saturated acids (C_8 to C_{20}). Acetone - water mixtures (45 to 90 per cent. of acetone) are used as eluting agents and the acids are titrated with 0·1 *N* alcoholic KOH to a green end-point (pH 6·8 to 7·0) of bromothymol blue. The dimensions of the column are 1·3 cm by 30 cm, and 2 to 50 mg of fatty acids are used. Myristic - linoleic acids and palmitic - oleic acids cannot be separated on the column; a preliminary oxidation with alkaline permanganate (Bertram) is required. The unsaturated acids are rendered insoluble in light petroleum (boiling range 40° to 60° C) or remain on the column. Yields of 95 to 98 per cent. for palmitic and 90 to 96 per cent. for myristic acid are attained. G. B. THACKRAY

2824. **The chromatographic separation of mixtures containing fatty acids, mono-, di- and triglycerides.** P. Savary and P. Desnuelle (*Bull. Soc. Chim. France*, 1954, **21** [7–8], 936–940).—The mixture, dissolved in ether, is first passed through a column of Amberlite IRA-400 resin, when the fatty acids are retained. The glycerides are then separated by reversed-phase partition chromatography on a column of silicone-treated kieselguhr, the solvents being *cyclo*hexane and aqueous ethanol. A trace of a mixture of glycerides of conjugated diene fatty acids is added to the mixture, and the concn. of glycerides in the chromatographic fractions can then be determined by measuring the absorption at 232 mμ. The isomerisation of the unstable 2-monoglycerides and 1:2-diglycerides can be avoided by substituting acetone for the ethanol in the partition chromatography. E. HAYES

2825. **Determination of sesamin, sesamolin and sesamol.** M. Beroza (*Anal. Chem.*, 1954, **26** [7], 1173–1176).—A method for the determination of sesamin, based on its separation in pure form by chromatography on silicic acid, is described. Solutions of ethyl acetate in 2:2:4-trimethylpentane are used to develop the chromatogram, and the sesamin in the effluent is detected and identified by means of its u.v. absorption. Sesamolin and sesamol are determined by the method of Suarez *et al.* (*Anal. Chem.*, 1952, **24**, 668). A soln. of known concn. is run simultaneously with the unknown, the concn. of which is found after comparison of the colours developed by the two solutions. Chromatographic studies indicate that sesamolin is the only compound, exclusive of sesamol, that gives the Villavecchia colour reaction. D. BAILEY

2826. **Measuring emulsion stability: a hydrometer method.** H. L. Sanders, H. R. Suter and E. R. Garverich (*Soap, N.Y.*, 1954, **30** [5], 99–103).—In emulsions of water and oil heavier than water, the density of the emulsion (measured by a hydrometer) progressively falls and approaches unity as the heavy oil globules settle downwards. Hence the stability of relatively unstable emulsions of this type can be characterised by the half-separation time, *i.e.*, the time taken for the density to fall halfway between its original value and unity. The hydrometer method is not practicable for lighter-than-water emulsions, as the surface cream formed when globules rise surrounds the hydrometer stem and causes spurious readings. G. HELMS

2827. **A rapid chromatographic method for the determination of vitamin A in whale-liver oils.** J. Green and D. O. Singleton (*Analyst*, 1954, **79**, 431–434).—The chromatographic medium used in the proposed rapid method for determining vitamin A in whole or saponified whale oils is floridin earth neutralised with NH_3. Unlike acid-washed floridin earth, this adsorbent quant. adsorbs vitamin A. When the non-saponified oil is used, the chromatographic column is neutralised by aspiration of NH_3 through it. It is then washed with *n*-hexane and the oil is introduced in this solvent. The column is eluted with *n*-hexane, an aliquot of the eluate is evaporated to dryness and the vitamin A is dissolved in *cyclo*hexane for spectrophotometric determination. In presence of significant amounts of anhydrovitamin A, the oil is saponified and the unsaponifiable portion is dissolved in *n*-hexane. The adsorbent column is neutralised with an ethanolic soln. of NH_3 and is washed with *n*-hexane. The unsaponifiable fraction of the oil is dissolved in *n*-hexane and passed into the column, which is then washed with *n*-hexane. Only anhydrovitamin A appears in the filtrate. The column is then eluted with a mixture of benzene and *n*-hexane (4 + 1), and an aliquot of the eluate is evaporated to dryness and dissolved in *cyclo*hexane as before. The method is not suitable for other oils, athough its use can lead to considerable elimination of interfering substances. A. O. JONES

2828. **Carotene and vitamin A in human fat.** A. W. Peirse (*Med. J. Aust.*, 1954, **i**, 589).—Samples are extracted with 30 per cent. v/v acetone in light petroleum for 1 hr.; total carotenoid pigment is determined spectrophotometrically at 450 mμ. An aliquot of the extract is chromatographed on a (1 + 1) mixture of MgO and Celite filter-aid and eluted with 10 per cent. v/v acetone in light petroleum. β-Carotene is also determined at 450 mμ. Another aliquot is chromatographed on a similar column, but is eluted only until β-carotene is observed to have reached the bottom of the column. All vitamin A is then assumed to have passed into the eluate. The solvent is evaporated and the residue is saponified with alcoholic KOH; the vitamin A is extracted with ether and determined by the Carr - Price reaction at 620 mμ, correction being made for the small amount of β-carotene that is still present. Two samples gave the following results: vitamin A, 2·2 and 2·0 μg per g; β-carotene, 6·1 and 5·1 μg per g. W. W. HOLLAND

2829. **Ash content of chick beak for vitamin-D assays.** Ling Wei, R. E. Pyke and D. B. Parrish (*J. Agric. Food Chem.*, 1954, **2** [11], 568–569).—The use of chick beak or toes, both of which are easily and rapidly prepared, is recommended for the biological determination of vitamin D in poultry-feed supplements instead of the tibia used in the A.O.A.C. method. Correlation between the two methods is close. S. C. JOLLY

2830. **A quick sulphide reduction of dehydroascorbic acid.** E. Pijanowski (*Bull. Acad. Polon. Sci.*, 1953, **1** [2], 73–77).—Dehydroascorbic acid can be conveniently reduced by means of acidified Na_2S and $HgCl_2$ solutions, and subsequently titrated with an indophenol dye. To 4 ml of

ascorbic acid solution (containing not more than 1·5 mg of vitamin) are added 1·4 to 1·5 ml of *N* HCl or H_2SO_4 and 0·7 ml of *M* Na_2S soln. The soln. is mixed and set aside for 10 to 15 min.; 1 ml of *M* $HgCl_2$ soln. is added, and the soln. is made up to 10 ml with H_2O, shaken and filtered. One to five-ml portions of the filtrate are titrated with 0·001 *N* 2:6-dichlorophenolindophenol until a permanent pink colour (15 sec. duration) is obtained (1 ml of 0·001 *N* indophenol dye ≡ 0·088 mg of ascorbic acid). The procedure may be applied to milk, ensilages and sauerkraut. G. R. WHALLEY

2831. **Radioactive tracer assay for cyanocobalamin and other cobalamins in complex mixtures.** F. A. Bacher, A. E. Boley and C. E. Shonk (*Anal. Chem.*, 1954, **26** [7], 1146–1149).—Cyanocobalamin (**I**) is assayed in complex mixtures ranging from fermentation products to vitamin capsules after its purification and concentration. The purified **I** is determined spectrophotometrically at 548, 430 and 361 mμ. **I** labelled with ^{60}Co is used as a tracer to determine recovery through the various operations necessary for purification. Other cobalamins are determined after conversion to **I**. Samples containing 100 μg of vitamin B_{12} at concn. as low as 0·1 μg per ml can be assayed. The standard deviation for duplicate assays is ± 4·3 per cent. D. BAILEY

2832. **On the supply and requirement of pyridoxine in pregnancy.** W. Neuweiler (*Schweiz. med. Wochschr.*, 1954, **84** [30], 883–884).—Pyridoxine excretion in the urine of pregnant women has been determined after a loading dose of 40 mg. Urine (2 ml) is placed in a 100-ml measuring flask and heated with 9 ml of HCl (2·5 per cent.) for 15 min. at 100° C. After cooling, 80 ml of distilled H_2O are added, the pH is brought to 9·0 by addition of $NaBO_2$ and made up to 100 ml with distilled water. The blue fluorescence is determined in a photo-electric colorimeter or in u.v. light. The standard is prepared with 0·4 μg of pyridoxine acid lactone. G. W. CAMBRIDGE

See also Abstract 2609.

Sanitation

2833. **Colorimetric determination of fluorine in waters and soil extracts.** W. M. Shaw (*Anal. Chem.*, 1954, **26** [7], 1212–1214).—Approx. 200 ml of a water sample (such as rain water or soil extract) are treated with 0·1 g of $CaSO_4$ and 0·5 g of decolorising carbon, neither of which should adsorb F′. To 100 ml of the clarified filtrate are added 5 ml of acid zirconyl-alizarin reagent (0·043 per cent. of $ZrOCl_2$ plus 0·007 per cent. of alizarin sulphonate, containing 3·7 per cent. v/v of H_2SO_4 and 11·2 per cent. v/v of HCl). The degree of colour discharge is compared at 0·2 p.p.m. intervals with standards containing 0 to 2 p.p.m. of F′ after development for 1 hr. The method is sensitive to 0·1 p.p.m. of F′. D. A. PANTONY

2834. **Colorimetric determination of manganese in waters.** E. Hluchán and J. Mayer (*Chem. Listy*, 1953, **47** [6], 846–849).—Addition of NaOH to the sample co-precipitates Mn as $MnO(OH)_2$ with $Mg(OH)_2$. Mg present in natural waters is usually sufficient, but to samples poor in Mg, add 0·5 ml of 30 per cent. aq. $MgSO_4$. The ppt. is dissolved in H_3PO_4, oxidised to Mn^{VII} with $K_2S_2O_8$ in the presence of $Ag^{\cdot}$ as catalyst, and Mn is determined colorimetrically. Addition of $Hg(NO_3)_2$ eliminates errors due to Cl′. Low results are obtained in the presence of $CaSO_4$. To the sample (up to 10 litres), add 8 per cent. NaOH to pH 12, decant the clear liquor, filter it, wash the ppt. with 4 per cent. NaOH, dissolve it on the filter in 20 per cent. H_3PO_4 (10 ml), and wash with water to make volume up to 30 ml. To the filtrate, add 0·5 per cent. Ag_2SO_4 (1 ml), a few drops of 10 per cent. $Hg(NO_3)_2$ and $K_2S_2O_8$ (0·1 g), heat on a steam-bath for 30 min., cool, add a crystal of $K_2S_2O_8$ and dilute with water to 50 ml. G. GLASER

2835. **Direct colorimetric method for the determination of chlorine dioxide in water.** H. W. Hodgden and R. S. Ingols (*Anal. Chem.*, 1954, **26** [7], 1224–1226).—Water (100 ml) containing up to 2·5 p.p.m. of ClO_2 is treated with 2 ml of tyrosine reagent (0·03 per cent. of tyrosine in sodium acetate buffer soln. at pH 4·5 to 4·6). The pink colour is measured by comparison with standard $Co(NO_3)_2$ solutions at ≃ 490 mμ [0·1 p.p.m. of ClO_2 ≡ 140 p.p.m. of $Co(NO_3)_2$]. Development time must be 8 to 10 min. Probable error is 2 to 12 per cent., depending on the ClO_2 concn. Chloramine, HOCl and $Mn^{\cdot\cdot}$ do not interfere. D. A. PANTONY

2836. **Quantitative determination of dissolved oxygen in nitrite-containing water using acid-chromous reagent.** H. W. Stone and P. Sigal (*Anal. Chem.*, 1954, **26** [7], 1236–1238).—A method for determining the amount of molecular O dissolved in nitrite-containing waters is described. The nitrites are destroyed with acid $KMnO_4$ solution, the excess of $KMnO_4$ is removed with oxalic acid and the residual O is determined with a mixture 0·05 *M* in $CrCl_2$ and 0·1 *M* in HCl. The method is rapid, gives reproducible results (av. deviation 0·25 per cent.) and has an accuracy exceeding that of the Winkler analysis. D. BAILEY

2837. **The effect of storage on coli-aerogenes and *Bacterium coli* counts of samples of non-chlorinated water-supplies.** S. B. Thomas (*Lab. Practice*, 1954, **3** [8], 331–333).—Results of recent studies on the effect of storage between collection and examination of water samples are reported and discussed. Overnight storage, even at 0° to 5° C, may cause significant changes in bacterial content and, owing to the decreased counts sometimes recorded, can result in acceptance of supplies that are not really safe. Effects of storage may depend on the nature of the water (surface or underground) and on the degree and time of bacterial pollution, which varies according to source (reservoir, river or farm supplies). Ingestion of bacteria by protozoa, destruction by antibiotics during storage, or the presence of Cu can give lower counts. Samples should be examined within 6 hr. after collection and should be kept at ≃ 5° C during transport. $Na_2S_2O_3$ in the sampling bottles inhibits any change in the real number of bacteria; 0·1 ml of aq. 3 per cent. $Na_2S_2O_3$ in a 6-oz bottle (0·3 ml in an 18-oz bottle) does not affect appreciably the counts of *B. coli* and coli-aerogenes of non-chlorinated water-supplies after storage for 6 hr. at 5° C. W. J. BAKER

2838. **Polarography of arsenic in the mineral waters of La Bourboule.** H. R. Olivier (*Bull. Soc. Chim. Biol.*, 1954, **36** [4–5], 695–703).—Conditions for polarographically determining small amounts of tervalent and quinquivalent As are investigated. A solution containing 10 ml of conc. HCl, 2 drops of 0·2 per cent. methylene blue, 50 ml of mineral water, and distilled water to 100 ml is de-gassed for

15 min. with N and polarographed with a drop time of 3 sec. The height of the first wave at −0·3 V is proportional to the concentration of As^{III}, which can be read off from a calibration curve. As^{V} is reduced by Na_2SO_3 - HCl, and SO_2 is removed by passing CO_2 for 1 hr. Total As is then determined as before. C. E. SEARLE

2839. **The molybdenum blue reaction and the determination of phosphorus in waters containing arsenic, silicon and germanium.** H. Levine, J. J. Rowe and F. S. Grimaldi (*Science*, 1954, **119**, 327–328).—Since As, Ge and Si also form heteropoly acids with Mo that yield blue compounds on reduction, a method is described for their removal before the estimation of P in sea-water. P in μg amounts can be estimated in the presence of at least 1 mg each of As_2O_3, GeO_2 and SiO_2 by concentrating it by pptn. with $Al(OH)_3$ followed by treatment of the ppt. with HF, HBr, HCl and H_2SO_4 to volatilise the As, Ge and Si. P is found to vary by the molybdenum blue reaction between 1·8 and 5·9 × 10^{-6} per cent. in sea-water.
G. M. LEWIS

2840. **"Incremental" spectrochemical analysis [of mineral waters].** V. Gazzi (*Chim. e Ind.*, 1954, **36** [4], 249–253).—The method of Duffendack and Wolfe (*Brit. Abstr. B*, 1938, 644) has been successfully applied to the determination of K or Li in mineral waters containing appreciable quantities of Ca, Mg and Na, with Mn as internal standard.
L. A. O'NEILL

2841. **Critical review of the literature of 1953 on sewage, waste treatment and water pollution. I. Analytical methods, sewage and radioactivity.** H. Heukelekian (*Sewage Ind. Wastes*, 1954, **26** [5], 573–615).—The analytical methods reviewed include the determination of B.O.D., O consumed, dissolved O, org. compounds, N compounds, metals, halogens, S, P and CN′ (64 references); the topics covered in the section on sewage include sedimentation and mechanical filtration, biological filters, activated sludge, disinfection, sludge digestion, disposal and utilisation, mechanical equipment, detergents, reclamation (166 references); the review of literature dealing with radioactivity deals with the radioactivity of rainfall, air, etc., permissible levels, decontamination, waste and sewage treatment, disposal and incineration and analytical methods (59 references). J. M. JACOBS

2842. **Fungi in polluted water and sewage. II. Isolation technique.** W. B. Cooke (*Sewage Ind. Wastes*, 1954, **26** [5], 661–674).—The effects of dyes, antibiotics and media on the development of mould populations in samples taken from 15 points in the Fairmount sewage treatment plant at Dayton, Ohio, ranging from the raw sewage after screening in detritus tanks to the pulverised heat dried sludge ready for packing and sale as a soil conditioner, are reported. Martin's original medium of acid, Rose Bengal and streptomycin (*Soil Sci.*, 1950, **69**, 215) was one of the more efficient media. Other media, although more efficient in permitting the growth of fungi, failed by allowing a large number of bacteria to develop. Most of the media tested allowed large mould colonies to develop, so preventing the growth of other moulds by competition. A satisfactory medium for the isolation of fungi from samples of sewage and of polluted water contains dextrose (10 g), protein hydrolysate (5 g), KH_2PO_4 (1 g), $MgSO_4$ (0·5 g), agar (20 g), and Rose Bengal (0·35 g per litre), with Aureomycin HCl (35 μg per ml). J. M. JACOBS

2843. **A chromatographic procedure for the determination of "pyrethrins" in pyrethrum extracts.** J. A. Cornelius (*Analyst*, 1954, **79**, 458–460).—A chromatographic method is described for the quant. separation of "pyrethrins" I and II; it is suitable for the assay of pyrethrum extracts in *n*-hexane. Extracts in mineral oil can be assayed after removal of the solvent. The activity of the Al_2O_3 used in the column is standardised by means of Sudan yellow and Sudan red, which are also used as marker dyes in the separation. The pyrethrum extract in *n*-hexane is added to the column with the mixed dyes soln. and the column is eluted with *n*-hexane containing 10 per cent. of ether. Collection of the "pyrethrin- I"eluate is started when the Sudan yellow reaches the base of the column, and the activity of the column should be adjusted so that with 70 to 95 ml of the eluate the Sudan red will have reached the base of the column. The eluate is finally adjusted to 100 ml. Elution of "pyrethrin II" is then started with *n*-hexane containing 20 per cent. of ether, 200 ml being collected. One-hundredth of each fraction is deprived of solvent and the residue is dissolved in aldehyde-free ethanol (20 ml). The optical density of the soln. is then determined at 224 mμ for "pyrethrin I" and 229 mμ for "pyrethrin II." $E_{1\,cm}$ × 17·85 = mg of "pyrethrin I" and $E_{1\,cm}$ × 20·39 = mg of "pyrethrin II" placed on the column. The factors are empirical, being based on parallel determinations by a mercury reduction method. A. O. JONES

2844. **Rapid colorimetric determination of total pyrethrins by reaction with sulphur.** L. W. Levy and R. E. Estrada (*J. Agric. Food Chem.*, 1954, **2** [12], 629–632).—A simple method is described for the determination of total pyrethrins (**I**) in extracts. To 1·0 ml of extract, or extract diluted if necessary with kerosine, containing 0·2 to 1·0 mg of **I** per ml in a test-tube, add 3·0 ml of S - CCl_4 reagent (2·50 g of flowers of sulphur dissolved in reagent grade CCl_4 without heating and diluted to 1 litre) and 3 ml of S - KOH reagent (0·25 g of flowers of sulphur dissolved without heating in 1 litre of approx. *N* anhydrous ethanolic KOH, and centrifuged if necessary with protection from air and moisture), and shake for 15 sec. Immediately place the stoppered tube in a water-bath at 30° ± 0·5° C for exactly 73 min. from the time of mixing, add a small amount of filter-aid, filter through a Whatman No. 1 paper (complete within 2 min.), shake the filtrate and, exactly 75 min. after mixing, measure the light absorption at 560 mμ against a blank of 1 ml of kerosine heated similarly. To correct for the original colour of the extract, repeat the above procedure, including the blank determination, using 3 ml each of CCl_4 and the KOH solution without the sulphur. From the difference between the two absorptions, derive the amount of **I** in the sample by comparison with the figure for a standard solution. For smaller amounts of **I** (0·04 to 0·2 mg), the light absorption should be measured at 440 mμ. Synergists and DDT do not interfere with the method. S. C. JOLLY

Agriculture and Plant Biochemistry

2845. **Polarographic determination of zinc in plant materials.** O. N. Hinsvark, W. H. Houff, S. H. Wittwer and H. M. Sell (*Anal. Chem.*, 1954, **26** [7], 1202–1204).—A sample of apple leaves (2 g) is ignited at 400° to 450° C for 12 hr., and the ash is dissolved in 2 ml of 6 *N* HCl. The soln. is evaporated to dryness and to the residue are added

2 ml of *M* tetrasodium ethylenediaminetetra-acetate. After evaporation of the solvent, 25 ml of 1·5 *N* NaOH are added, and the ZnO_2'' in the filtered soln. is measured amperometrically at an applied $E_{\frac{1}{2}}$ of 1·32 ± 0·02 V. The step height is compared with a reference curve prepared from standards that have been treated similarly. Comparison is made with the dithizone colour method, with respect to which results are consistently slightly low. D. A. PANTONY

2846. **The estimation of carbohydrates in plant extracts by anthrone.** E. W. Yemm and A. J. Willis (*Biochem. J.*, 1954, **57** [3], 508–514).—The anthrone colour reaction is investigated with a wide range of naturally occurring pentoses and hexoses. Fructose and sorbose react most rapidly, to give max. colour development after ≃ 1·5 min. Aldoses react more slowly and give much less colour, being max. after 6 to 7 min. for galactose and mannose, and ≃ 10 min. for glucose. Rhamnose and fucose are very similar with regard to colour production, and max. development occurs after 3 min. Xylose, ribose and arabinose give much less colour than do the aldohexoses, and max. colour is formed in 2 to 2·5 min. Xylose gives the most intense colour. With all sugars examined, the colour decreases with further heating. With pentoses and hexoses, the max. optical density occurs at ≃ 630 mμ. Mixtures of sugars give results that agree closely with those expected from the colour production of the individual sugars. Under the given conditions there is no evidence of serious inter-action of hexoses and pentoses. There is increased colour production in presence of chlorides, but other compounds cause little interference. Uronic acids give a small, but definite, colour formation. The anthrone method is applicable to the sugars of stable glycosides which may constitute much of the soluble carbohydrate material in some plant tissues. Used in conjunction with chromatographic identification, the method is of value when limited amounts of tissue are available. J. N. ASHLEY

2847. **Detection of glycosides and other carbohydrate compounds on paper chromatograms.** J. A. Cifonelli and F. Smith (*Anal. Chem.*, 1954, **26** [7], 1132–1134).—A method is described for the detection of non-reducing glycosides on paper chromatograms. The dried chromatograms are lightly sprayed with a saturated aq. soln. of KIO_4 followed by a benzidine soln. after 2 to 6 min. The glycosides, which react with periodates, are located by the appearance of colourless spots on a blue background, arising from the action of the periodate on benzidine. The method, which is fairly sensitive, is suitable for the detection of other carbohydrate compounds capable of being oxidised by periodate. A method of differentiating between certain carbohydrate compounds is suggested; it is based upon the rate of reaction with periodate. D. BAILEY

2848. **Separation of ribonucleic acid and deoxyribonucleic acid by electrophoresis. (Detection of ribonucleic acids by marking with ^{32}P).** M. Deimel (*Biochem. Z.*, 1954, **325** [5], 358–365).—A method is described for the separation and identification of ribonucleic acid and deoxyribonucleic acid by paper-electrophoresis. The nucleic acids were marked *in vivo* with ^{32}P by subcutaneous administration of inorganic ^{32}P (1 to 4 mc) to rats 24 hr. before the preparation of extracts of liver, thymus and spleen. Yeast nucleic acids were also marked *in vivo* before extraction. Both acid and alkaline extracts were prepared and have been compared. The electrophoretic separation was on filter-paper strips with 0·01 to 0·03 ml of extract, containing 10 to 30 μg of nucleic acid. Barbitone buffer, pH 8·6, was used, a potential of 200 V being applied for 5 to 6 hr. The sites of radioactivity were determined with a Geiger - Müller counter and were confirmed by comparison with chemically separated ribonucleic acids, by selective enzymatic destruction with a specific ribonuclease and by development of the spots with Pyronine - methyl green. Ribonucleic acid gave a red spot and deoxyribonucleic acid a blue-green spot. Deoxyribonucleic acid moves at about 1·4 times the speed of ribonucleic acid and at twice the speed of human serum albumin. G. W. CAMBRIDGE

2849. **The determination of functional groups in tannins and lignins. I. Determination of hydroxyl groups.** W. E. Hillis (*J. Soc. Leath. Tr. Chem.*, 1954, **38** [6], 177–183).—The accuracy of a method for determining the total hydroxyl content of tannins and lignins is fully investigated. Details of the method are fuller than those given in the report by Buchanan *et al.* (*J. Amer. Leath. Chem. Ass.*, 1950, **45**, 513). Recoveries for hydroxyl groups were as follows: pyrogallol, 99·7 per cent.; phloroglucinol, 99·0 per cent.; eugenol, 98·8 per cent.; catechin 98 per cent.; and aromadendrin, 100·4 per cent. Results for lignin were consistent, but those for vanillin were variable. B. R. HAZEL

2850. **Studies on saponins. V. Analytical method for differentiating steroid and triterpenoid saponins.** J. L. Fontán-Candela (*An. Soc. Esp. Fis. Quim., B*, 1954, **50** [4], 441–444).—The aq. vegetable extract is treated with CCl_4 to remove fats or chlorophyll, and foaming tests (*An. Soc. Esp. Fis. Quim., B*, 1953, **49**, 325) are carried out on the aq. residue at pH 1 and pH 13 at constant pressure. If the foaming time is similar at both pH values, the saponin is triterpenoid; if the time is much greater at pH 13, the saponin is steroid. L. A. O'NEILL

2851. **Estimation of available phosphorus in soils by extraction with sodium bicarbonate.** S. R. Olsen, C. V. Cole, F. S. Watanabe and L. A. Dean (*U.S. Dep. Agric. Circ. No.* 939, 1954, 19 pp.).—A new method for extracting soil P with 0·5 *M* $NaHCO_3$ at pH 8·5 is described. Five g of soil are shaken with 100 ml of the reagent plus 1 teaspoonful of carbon black (Darco G-60) for 30 min., and filtered. P is determined in 5 ml of the filtrate by Dickman and Bray's method (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 665). E. G. BRICKELL

2852. **Colorimetric estimation of malathion residues.** M. V. Norris, W. A. Vail and P. R. Averell (*J. Agric. Food Chem.*, 1954, **2** [11], 570–573).—A colorimetric method is described for the determination of S-(1:2-diethoxycarbonylethyl)-O:O-dimethyl dithiophosphate (malathion) (**I**) on a wide variety of plant materials. Agitate continuously for 4 hr. 0·5 to 1·0 kg of the disintegrated sample with a measured vol. of CCl_4 (1 to 3 ml per g of sample) in a closed vessel, allow to separate, adjust the volume of an aliquot of the CCl_4 layer containing 0·25 to 2·5 mg of **I** to 100 ml either by diluting with CCl_4 or by evaporating on a water-bath, add 25 ml of pure ethanol and 1 ml of 6 *N* NaOH, and shake vigorously for exactly 1 min. The remainder of the determination must be completed without delay within 1 hr. Immediately add 75 ml of 2 per cent. NaCl solution, cooled to ≃ 15° C, shake for

exactly 1 min., allow to separate and discard the CCl_4 layer. Add 25 ml of CCl_4, shake vigorously for 30 sec., allow to separate, and discard the CCl_4 layer together with any small volume of emulsion and suspended solids. Add 1 ml of 7 *N* HCl, and extract with 25-ml portions of CCl_4 until no more yellow colour is extracted; completely remove the last extract and discard the extracts. Add 25 ml of CCl_4 and 2 ml of 1 per cent. $CuSO_4$ solution, shake vigorously for 1 min., and allow to separate. Immediately measure the yellow colour of the CCl_4 layer in a spectrophotometer or photo-electric colorimeter at 418 mμ against CCl_4 as a blank. Derive the amount of **I** present by reference to a standard graph prepared by putting 0 to 25-ml portions of a standard solution (0·1 mg of **I** per ml) through the same procedure. S. C. JOLLY

2853. **Chemical determination of aldrin in crop materials.** A. E. O'Donnell, M. M. Neal, F. T. Weiss, J. M. Bann, T. J. DeCino and S. C. Lau (*J. Agric. Food Chem.*, 1954, **2** [11], 573–580).—Two methods are described for the determination of 1:2:3:4:10:10-hexachloro-1:4:4a:5:8:8a-hexahydro-1:4-5:8-di*endo*methylenenaphthalene (aldrin) (**I**) residues of the order of 0·1 p.p.m. in plant materials. Extract the disintegrated material by shaking it for 1 hr. with Skellysolve B (2 ml per g for most samples of low water content, 6 ml per g for lucerne, clover, silage, foliage, hay, grass, tobacco, etc., and 2 ml plus 0·3 ml of *iso*propanol per g for samples of high water content). Decant the liquid through a filter-paper and, if alcohol has been used, wash the filtrate with 3 portions of water, each equal to the volume of alcohol used. Dry the extract over anhydrous Na_2SO_4 and filter it. Continue by one of the following procedures. (*i*) Evaporate an aliquot to dryness, saponify the residue by refluxing it for 1 hr. with ≮ 50 ml (*V*) of alcoholic KOH solution (0·25 g in 3 ml per g of residue), add *V* ml of water, extract with six *V*-ml portions of Skellysolve B, wash the combined extracts with several portions of water, dry them over anhydrous Na_2SO_4, filter, and evaporate to 100 ml. (*ii*) If **I** is determined finally by combustion method (*a*) [below], concentrate an aliquot of the filtered extract equiv. to 200 to 300 g of sample to 100 ml. (*iii*) For fruits and most vegetable in which **I** is finally determined photometrically [see (*b*) below], concentrate an aliquot of the filtered extract equiv. to 100 to 200 g of sample to 100 ml. For grasses, forage crops and vegetables, shake an aliquot of the filtered extract equiv. to 100 g of sample with 30 g of a mixture of activated carbon, silicic acid and a filter aid for 1 to 2 min., decant through a filter-paper, wash the residue and filter with five 50-ml portions of Skellysolve B, and evaporate the combined filtrate and washings to 100 ml. Pass the 100 ml of conc. extract from (*i*), (*ii*) or (*iii*) through a column of the adsorbent mixture used in (*iii*) packed between two shallow layers of anhydrous Na_2SO_4, and wash the extract through with 120 ml of solvent. Determine **I** in the eluate either by (*a*) adding 100 mg of Cl-free white oil, evaporating the solvent and determining the Cl in the residue by the amperometric titration procedure of Agazzi, Peters and Brooks (*Brit. Abstr. C*, 1953, 367), or (*b*) an improved modification of the phenyl azide photometric method of Danish and Lidov (*Brit. Abstr. C*, 1950, 405). Apparent **I** values of ≮ 0·08 and 0·05 p.p.m. are obtained by (*a*) and (*b*), respectively, on **I**-free materials, and recoveries are accurate to less than 0·1 p.p.m.
S. C. JOLLY

2854. **Application of chromatography in determination of micro quantities of 3-(*p*-chlorophenyl)-1:1-dimethylurea.** W. E. Bleidner (*J. Agric. Food Chem.*, 1954, **2** [13], 682–684).—In the micro-determination of N′-(*p*-chlorophenyl)-NN-dimethylurea in plant tissues by hydrolysis to *p*-chloroaniline (**I**) and subsequent conversion to an azo-dye, the interference caused by the formation of traces of aromatic amines, chiefly *o*-aminoacetophenone (**II**), from plant tissues during the hydrolysis can be eliminated by including a chromatographic purification stage in the standard procedure. The mixture of azo-dyes is poured on to a column (15 mm in diameter, 20 to 25 cm long) of dry cellulose (Whatman No. 1 filter-paper), and the chromatogram is developed with *N* HCl, when the dye due to **II** is eluted first. The magenta-coloured dye due to **I** is then eluted with a mixture of *N* HCl and glacial acetic acid (1 + 1), the band usually passing out of the column in a 10 to 15-ml fraction, which is then diluted if necessary and its light absorption measured. The presence of acetic acid does not affect the hue or intensity of the colour. With this method it is not necessary to analyse a corresponding sample of untreated material to correct for interfering substances. S. C. JOLLY

2855. **Detection of internal insect infestation in grain by sound amplification.** R. E. Adams, J. E. Wolfe, Max Milner and J. A. Shellenberger (*Cereal Chem.*, 1954, **31** [3], 271–276).—An electronic amplification system is described for the detection of sounds produced by insects, such as the granary weevil and rice weevil, infecting wheat internally. Applications include evaluation of fumigant effectiveness and monitoring infestation in storage bins. S. C. JOLLY

See also Abstracts 2843, 2844.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

2856. **Micro-determination of nitrogen with the aid of diffusion cells. (A simple apparatus for isothermal distillation at high temperature.)** H. Wüst (*Klin. Wochschr.*, 1954, **32** [27–28], 660–661).—The construction of a micro-diffusion cell in Jena glass for determining N is described. By using $(NH_4)_2SO_4$ (212 μg of N) as test soln. and applying the usual Kjeldahl technique, 97 to 98 per cent. of the NH_3 was distilled over in 5 hr. at 80° C, whereas only 77 per cent. was distilled over in 15 hr. at 4° C. The diffusion kinetics of the cell are discussed. G. W. CAMBRIDGE

2857. **Apparatus for the micro-analytical determination of C-methyl and acetyl groups.** E. Wiesenberger (*Mikrochim. Acta*, 1954, [1], 127–139).—An apparatus for the micro-determination of acetyl groups is described. It can be used to carry out acid or alkaline hydrolyses, and for determination of C-methyl groups by the oxidation process with chromic acid and by esterification. To destroy decomposition products, which are formed to a considerable extent during acid and alkaline hydrolysis, the vapours evolved are passed through hot chromic acid - sulphuric acid. Accurate results were obtained by the method with octa-acetylcellobiose by acid or alkaline hydrolysis; with penta-acetylglucose, results were accurate only when alkaline hydrolysis was used. A. J. MEE

2858. **Automatic sediment and moisture recorder.** E. B. Delgass, J. M. Brooks, S. Kleinheksel and A. E. Traver (*Ind. Eng. Chem.*, 1954, **46** [7], 1418–1422).—Suspended material and water in piped petroleum products are recorded by withdrawing a small sample, which is passed through a continuously moving strip of filter-paper. The sediment is estimated by visual comparison of the trace with a set of standards. Moisture is measured by the resistance of the filter-paper. G. SKIRROW

2859. **An automatic device for starting paper chromatograms.** L. J. B. Husband (*Chem. & Ind.*, 1954, [27], 776–777).—An automatic device is described and sketched; it starts chromatograms by bringing solvent into contact with the prepared filter-paper at a pre-determined time during the night so that the chromatograms are ready for examination in the morning. Equilibration of solvent and vapour is established during a period of about 6 hr. before the solvent is allowed to run into the prepared empty trough. I. JONES

2860. **Modified horizontal migration method in paper chromatography.** N. C. Ganguli (*Nature*, 1954, **174**, 189–190).—A modification of the chromatographic technique described by Matthias (*Naturwiss.*, 1954, **41**, 17) is suggested. The method consists in a combination of circular and unidimensional chromatography. A filter-paper (Whatman No. 1) 18·5 × 18·5 cm or 18·5 cm in diam. is prepared as follows. Across the diameter of the paper and to within 3 cm of the edge a series of holes are cut 1 cm apart to take paper wicks (2·0 × 1·5 cm); along a line parallel to this, two series of slots (width 2 mm) are cut on either side of the row of holes and 0·5 cm away from it. Between each slot is a 2-mm gap, directly opposite the wick hole. Two long cuts are made at the end of the row of holes, and at right-angles to it, extending to within 2 cm of the periphery of the paper. The material to be chromatographed is placed at the mid-point of the gap in the row of slots. The final arrangement is the equivalent of 22 1-cm paper strips, development taking place radially at first. The improvement in resolution over simple unidimensional technique with mixtures of amino-acids is demonstrated and a large number of samples can be run under identical conditions. G. B. THACKRAY

2861. **Methods and instrumentation for air sampling.** L. F. Dieringer and W. T. Ingram (*Instruments & Automation*, 1954, **27** [7], 1086–1089).—Methods of sampling air for the determination of particulate matter and gases are reviewed. Recent advances made possible by photo-electric devices and chemical instrumentation are described. G. SKIRROW

2862. **Lunge nitrometers.** British Standards Institution (B.S.2070:1954, 12 pp.).—Specifications are given of three sizes of nitrometer of total capacities 50, 100 and 130 ml suitable for estimation of N in nitrates. G. SKIRROW

2863. **A simple apparatus for determining carbon dioxide in small samples.** C. F. M. Fryd (*Lab. Practice*, 1954, **3** [8], 333–334).—The apparatus described and illustrated is used for routine estimations of CO_2 in mg samples, such as drillings from bones or teeth; results with an error of about ± 1 per cent. can be obtained in 10 min. The device is made by modifying a ground-glass joint (B.S. 572:1950), to which is sealed via a constriction a capillary glass-tube (12 cm by 1 to 2 mm) containing a drop of Hg as vol. indicator. The tube rests horizontally by means of four attached glass-legs. The sample is placed on a platform cut in the male half of the joint, and the acid is put in a cell formed by rounding-off the female half. The two halves are tightly joined, and the initial and final positions of the Hg are noted (tube horizontal), the reaction being started by tilting. The tube is calibrated with carbonates of known CO_2 content. W. J. BAKER

2864. **Determination of sulphur trioxide in gases. The efficiency of a sintered-glass filter in recovering sulphuric acid mist.** A. W. Fletcher (*Chem. & Ind.*, 1954, [27], 777–778).—The efficiency of a grade-4 sintered-glass filter in recovering H_2SO_4 mist during the determination of traces of SO_3 in the presence of SO_2 in gases (Corbett, *Brit. Abstr. C*, 1952, 47) is examined and found to be satisfactory. A diagram of the apparatus is given. I. JONES

2865. **A statistical study of the performance of an Ainsworth microchemical balance.** J. T. Waber and G. E. Sturdy (*Anal. Chem.*, 1954, **26** [7], 1177–1180).—By statistically examining the results of repeated weighings of samples of Pt, Au and Al against gold weights under controlled conditions of humidity within the room and the balance case, possible correlations between specimen weight and temp. and humidity have been sought. The results indicate that the apparent weight of an object does not change significantly with slow changes in the environment. G. SKIRROW

2866. **Pregl precision weight pipette for the determination of the specific gravities of liquids.** A. Haack and G. Wieser (*Mikrochim. Acta*, 1954, [1], 117–121).—The suitability of the Pregl weighing pipette for the determination of the specific gravity of a liquid has been investigated. Evaporation, which is reduced when the discharge tips are not too narrow, can be prevented almost completely if a narrow tube provided with several capillaries is fastened to the tip by a short length of rubber tubing. This apparatus is then the most accurate available for the determination of the densities of liquids. A. J. MEE

2867. **A modified weight pipette.** M. A. Birch (*Chem. & Ind.*, 1954, [27], 776).—The Lunge - Rey weight pipette has been modified so as to make it more generally useful for the weighing of liquid samples. The modified form (illustrated) is especially useful when a number of weighings has to be made on one sample. I. JONES

2868. **Pyknometer method of determining density of viscous, plastic or solid materials.** R. Hock (*Prakt. Chem.*, 1954, **5**, 110–111).—The pyknometer is made by sealing one end of a glass tube 3 to 5 cm long and 0·4 to 1 cm in diameter, and grinding plane the edge of the other end on a carborundum wheel so that it can be closed by a microscope cover glass held in place by atm. pressure. For viscous or pasty fluids, the bottle is half filled with the material and the whole is weighed. The material is carefully melted, air bubbles are removed, and the tube is filled with water and reweighed. For substances soluble in water, a non-solvent liquid is used. The density is calculated in the usual way. A. R. PEARSON

2869. **Report on recommended specifications for microchemical apparatus (1953). Weighing and drying.** Committee on Microchemical Apparatus.

Division of Analytical Chemistry, American Chemical Society (*Anal. Chem.*, 1954, **26** [7], 1186–1190).—The report includes specifications for combustion boats, weighing bottles, metal spatulas, forceps and tare flasks. A modified Abderhalden drying apparatus is also described. G. SKIRROW

2870. **Improvements relating to apparatus for determining particle size distribution.** Simon-Carves Ltd. and W. Bostock (Brit. Pat. 712,434; Date Appl. 3.11.52).—A settling-tube containing a suspension of the finely divided material in a liquid has its lower end open and immersed in a bulk of the same liquid. The settled particles in a receiver under the lower end of the tube may be continuously weighed by an attached torsion balance. A pre-mixing tube is attached to the top of the settling tube, which is enclosed in a constant-temp. jacket. The apparatus is principally intended for particles of sizes 5 to 75 μ. D. R. GLASSON

See also Abstracts 2671, 2737, 2738.

Optical

2871. **Quantitative spectrochemical analysis with the aid of a slitless spectrograph.** A. Cornu (*Cahiers de Phys.*, 1953, No. 45, 28 pp.).—Two juxtaposed spectra are produced from two sources fed by one Feussner generator, one of the sources having a standard-alloy electrode. The distance of one source is altered until the line intensities of the two spectra become equal. Analyses of a number of Cu - Zn standard alloys have been checked. R. B. CLARKE

2872. **Spectrophotometers [for measuring changes in light intensity throughout a spectrum].** National Research Development Corp. (Brit. Pat. 712,092; Date Appl. 20.4.51).—A spectrophotometer is described in which the light beam can be modified in its spectral composition and developed into a spectrogram, having an interfering beam superimposed on it. The difference in the optical paths of the beams is varied along the length of the colour lines of the spectrogram to set up dark fringes transverse to the colour lines; one of the beams can also be subjected to an intensity variation along the length of the colour lines. A double-beam interferometer is combined with a wedge spectrograph, the wedge being disposed in one beam of the interferometer; provision is made for the inclusion of an object under investigation in the other beam and for phase compensation for the wedge and object in the respective beams. The spectrograph advantageously comprises a Féry prism with aplanatic spherical surfaces. The apparatus eliminates inaccuracy of wedge spectrograph measurements caused by imperfections in the photographic process. D. R. GLASSON

2873. **Thermostat cell-holder for Beckman spectrophotometer.** M. Dixon (*Biochem. J.*, 1954, **58** [1], 1–3).—The cell-holder and its construction are described. It consists essentially in a Perspex box surrounding two standard 10-mm quartz cells. The temp. of the cells is kept constant by water circulating in contact with them. This gives rapid equilibration and accurate control. Water-tight joints between the cells and the box are made by compressed rubber gaskets. J. N. ASHLEY

2874. **Beckman flow colorimeter.** J. F. Bishop and R. S. White (*Ind. Eng. Chem.*, 1954, **46** [7], 1432–1435).—A light beam is passed through a known amount of the continuously flowing process fluid and suitable filters. The transmitted light is observed by a photo-tube, the output of which is amplified and recorded. G. SKIRROW

2875. **Ultra-violet spectrophotometer for automatic control.** G. G. Campbell and J. B. Godin (*Ind. Eng. Chem.*, 1954, **46** [7], 1413–1417).—Details are given of the application of Beckman Model DUR recording u.v. spectrophotometer as an automatic control in a plant producing 1:3-butadiene and *n*-butylenes. G. SKIRROW

2876. **Improvements in or relating to methods and means for testing a translucent fluid substance.** E. F. Daly and Unicam Instruments (Cambridge) Ltd. (Brit. Pat. 710,867; Date Appl. 2.4.52).—The absorption spectrum of the test translucent fluid is continuously compared with that of a known reference specimen. The apparatus comprises means for passing a beam of light to a photo-cell or other light detector through a monochromator in series with it; alternatively it may be passed through a body of fluid of known composition, to produce a detector output having an alternating voltage component corresponding to the absorption difference of the two specimens at the wavelength passed by the monochromator. The latter is caused periodically to perform spectral sweeps through a pre-determined spectrum range, and is synchronised with a time-base sweep applied to the beam of a cathode-ray tube with a deflection transversely to the time-base sweep according to the amplitude of the alternating voltage component. A claim is also made for a method of scanning wavelengths in plotting spectra. Fast scanning is permitted by using a photomultiplier in the visible and u.v. and a photo-conductive cell in the near i.r. Possible modifications of the apparatus mentioned include application to gas analysis and automatic balancing of the spectra by servo-mechanism control of the variable-path absorption cells. D. R. GLASSON

2877. **Recording colorimetric, nephelometric and fluorimetric measurements during automatic continuous dilution.** H. J. O. Breitner (*Kolloidzschr.*, 1954, **136** [2–3], 155–156).—Continuous automatic dilution and recording are used to investigate the effects of concentration on the light absorption, turbidity and fluorescence of solutions. Advantages include increased accuracy and the possibility of investigating the effects of age and other variables. A. B. DENSHAM

2878. **A simple variable-thickness infra-red absorption cell.** B. M. Mitzner and S. Z. Lewin (*J. Opt. Soc. Amer.*, 1954, **44** [5], 425–426).—The cell described has silver chloride windows separated by a polythene spacer. By filling the cell from a syringe, the applied pressure causes the two windows to separate to many times the thickness of the spacer owing to the flexibility of silver chloride. The required thickness is then attained by compressing the windows with a micrometer screw to the necessary degree. B. S. COOPER

2879. **Sensitising non-dispersive infra-red analyser.** E. H. Woodhull, E. H. Siegler and H. Sobcov (*Ind. Eng. Chem.*, 1954, **46** [7], 1396–1400).—A specific non-dispersive infra-red analyser is described; it is calibrated in percentage concn. of one component in a multi-component gas stream.

The method of calibration described can be applied to non-dispersive analysers by use of either selective or total radiation type detectors. G. SKIRROW

2880. **A reflecting microscope for infra-red absorption measurements.** K. P. Norris (*J. Sci. Instrum.*, 1954, **31** [8], 284–287).—Factors influencing the design of objectives for infra-red microspectrometers are discussed. A bi-spherical objective of numerical aperture 0·8 with a central obstruction of 40 per cent. is shown to be suitable for the rock-salt region. The dimensions of 3 objectives are given together with details of construction. G. SKIRROW

2881. **Spectroscopy from the point of view of communication theory. IV. Automatic recording of infra-red spectra on punched cards.** G. W. King, E. H. Blanton and J. Frawley (*J. Opt. Soc. Amer.*, 1954, **44** [5], 397–402).—The advantages are described of incorporating a digital reader in parallel with the conventional graphical recorder of an infra-red spectrometer. By the use of this equipment, spectroscopic information may be transferred to punched cards. This leads to automatic machine methods of processing data from calibration, conversion to absorption coefficient, comparison of spectra, or subtraction of spectra. B. S. COOPER

2882. **Field application of infra-red analysers.** R. F. Wall, A. L. Giusti, J. W. Fitzpatrick and C. E. Wood (*Ind. Eng. Chem.*, 1954, **46** [7], 1387–1390).—The infra-red instrumentation of an acrylonitrile plant for continuous analytical control is described. G. SKIRROW

2883. **Installation of continuous infra-red analysers. Sample treatment.** S. H. Walters (*Ind. Eng. Chem.*, 1954, **46** [7], 1390–1393).—Considerations involved in the application of absorption instruments to plant stream control are reviewed. G. SKIRROW

2884. **Infra-red gas analyser for butane splitter control.** R. L. Martin and B. W. Thomas (*Ind. Eng. Chem.*, 1954, **46** [7], 1393–1396).—A description is given of the application of a non-dispersive infra-red gas analyser for the continuous measurement of the *iso*butane losses from three parallel operating butane splitter columns. G. SKIRROW

2885. **Application of bichromator infra-red dispersion analyser.** A. Savitzky and D. R. Bresky (*Ind. Eng. Chem.*, 1954, **46** [7], 1382–1386).—The ratio of transmission of flowing samples is measured at two wavelengths, one of which is chosen at an absorption maximum. The other reference wavelength is selected to eliminate interference by other components of the stream. A servo operated wedge enables optical null balance to be maintained. Applications to gaseous and liquid systems are described. G. SKIRROW

2886. **Infra-red analyser for monitoring water content. Liquid sulphur dioxide - gas oil extraction unit.** F. W. Karasek and E. C. Miller (*Ind. Eng. Chem.*, 1954, **46** [7], 1374–1376).—An infra-red analyser is described for the measurement of the water content (2·7-μ band) in liquid SO_2. The instrument has a full-scale span of 0·4 per cent. w/w of H_2O with an accuracy of $\pm$ 0·01 per cent. w/w. It has been applied as a continuous water-content indicator in a refinery SO_2 - gas oil extraction unit as an aid to protection against corrosion. G. SKIRROW

2887. **Fluoroscope and Geiger counter for measuring ultra-violet absorption of chromatograms.** T. D. Price and P. B. Hudson (*Anal. Chem.*, 1954, **26** [7], 1127–1132).—A halophosphate fluoroscope and a Geiger counter with a stainless steel cathode were found to be sensitive to radiation in the far u.v., and have been used as detectors in densitometric instruments for the measurement of the absorption of chromatograms. G. SKIRROW

2888. **Recording differential refractometer. Continuous plant stream monitoring.** D. N. Campbell, C. G. Fellows, S. B. Spracklen and C. F. Hwang (*Ind. Eng. Chem.*, 1954, **46** [7], 1409–1412).—The refractometer described incorporates a transistor amplifier having a gain of approx. 40,000 and has a sensitivity of 3×10^{-6} refractive index units. G. SKIRROW

2889. **A new optical system for simultaneous recording of refractive index and its gradient in stratified solutions.** H. Svensson and R. Forsberg (*J. Opt. Soc. Amer.*, 1954, **44** [5], 414–416).—A hollow prismatic cell can be combined with the astigmatic schlieren arrangement so that a refractivity curve and a gradient curve are superimposed on the one record. B. S. COOPER

2890. **Dispersional light filters of high monochromaticity.** F. A. Korolev and A. Yu. Klement'eva (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1954, **94** [6], 1025–1027).—The band width of the light transmitted by a dispersional light filter, composed of finely ground glass immersed in a liquid, can be decreased (at the expense of the percentage transmission) by combining two filters, having the same wavelength for max. transmission but with slightly overlapping transmission curves. R. C. MURRAY

2891. **An apparatus for the determination of molecular weights by light-scattering.** L. Harvey and D. Cleverdon (*J. Sci. Instrum.*, 1954, **31** [8], 274–279).—Constructional details are given of a light-scattering apparatus that includes a cell designed to minimise unwanted reflections. The scattered light is observed through plain windows that are axially opposite to light-trap cones. The metal cell is blackened internally. G. SKIRROW

2892. **Liquid scintillation counting of tritium and ^{14}C-labelled compounds.** D. J. Rosenthal and H. O. Anger (*Rev. Sci. Instrum.*, 1954, **25** [7], 670–674).—The radiations from solutions of the active materials in xylene containing *p*-terphenyl- and diphenylhexatrienes are detected by a cooled photo-multiplier tube. A minimum of $4{\cdot}3 \times 10^{-10}$ curies of ^{3}H or $1{\cdot}8 \times 10^{-11}$ curies of ^{14}C can be measured. G. SKIRROW

Thermal

2893. **Laboratory thermometers.** British Standards Institution (B.S. 593:1954, 24 pp.).—Four series of thermometers are specified. *Series A*—Range of 30° to 40° C and graduated at each 0·1°. *Series B*—Range of about 60° C graduated at each 0·2°. *Series C*—Thermometers without zeros covering ranges of 100° C and graduated at

each 1°. *Series F*—Thermometers covering various ranges between −2° and + 400° C. In each of the above series are included the equivalent Fahrenheit thermometers. G. SKIRROW

2894. **Incubator, water-bath and oven thermometers for laboratory use.** British Standards Institution (B.S.619:1954, 9 pp.).—Specifications are given for four mercury-in-glass solid-stem thermometers having ranges 20° to 60° C, 30° to 75° C, 65° to 125° C and 115° to 180° C. The thermometers can be read to about ± 0·1° C. G. SKIRROW

2895. **Measuring fine temperature changes.** L. G. R. Sims (*Engineering, Lond.*, 1954, **177**, 15–16).—The problems of accurately calibrating thermocouples and of measuring very small changes in temp. as required in calorimetry are discussed. When a reflecting coil galvanometer and amplifier are used, the lowest input level is 0·25 microvolt or 0·006° C with a base-metal couple; if a chopping amplifier is introduced, 0·01 microvolt or 0·00025° C can be measured. Various methods of obtaining a stable cold junction, necessary for high accuracy at fine temp. changes, are described; the triple-point water cell is probably the ideal solution for long-period work and gives a long-term absolute temp. value of 0·01 ± 0·0001° C. B. C. U. R. A.

2896. **Heating and cooling blocks for microchemical purposes.** British Standards Institution (B.S.1428:Part G1:1954, 13 pp.).—Aluminium or light-alloy heating and cooling blocks for use with microchemical combustion boats, crucibles, beakers and centifruge tubes are specified. G. SKIRROW

Electrical

2897. **Improvements in electrolytic determination of the concentration of a constituent in a solution.** Leeds and Northrup Co. (Brit. Pat. 711,812; Date Appl. 6.11.51).—The methods and apparatus described are applied particularly to the determination of halogens. An improved control system is provided by means of which the titrating current in a continuous system is always indicative of the concn. of the constituent in the electrolyte. Determinations of Cl at pH 0·3 to 1·8 in H_2SO_4 are described, Fe^{+++} (0·04 to 5 per cent.) being used as electro-catalytic agent in the reduction of Cl to Cl′. D. R. GLASSON

2898. **Shielded dropping-mercury electrode for polarographic analysis of flowing solutions. Determination of cyanide ion.** W. H. Jura (*Anal. Chem.*, 1954, **26** [7], 1121–1123).—Details are given for the construction and manipulation of a glass-shielded mercury electrode (Laitinen and Burdett, *Brit. Abstr. C*, 1950, 468) that can be used for polarographic examination of flowing solutions. The apparatus is tested for reproducibility and response time with oxygen-saturated aq. solutions of KCN (0·001 to 0·01 *M* in 0·25 *M* NaCl and 0·02 *M* NaOH). Results agree well with those by the "static" method, and reproducibility is within 1 to 2 per cent. Response time is rapid, 98 per cent. equilibrium being reached within 2 min. D. A. PANTONY

2899. **The effect of cracked cells on the shape of cathode-ray polarograph traces.** G. F. Reynolds and H. I. Shalgosky (*Chem. & Ind.*, 1954, [27], 787).—A straight line extending diagonally across a cathode-ray tube screen, instead of a polarograph step of normal shape with linear sweep cathode-ray polarographs, is shown to be caused by the use of a polarographic cell with a crack in the glass wall. I. JONES

2900. **Potentiometric instrument for sulphur determination [in gases].** H. Landsberg and E. E. Escher (*Ind. Eng. Chem.*, 1954, **46** [7], 1422–1428).—An automatic instrument (the Titrilog) is described for continuously recording trace quantities of oxidisable sulphur compounds in the range 0·1 to several hundred p.p.m. by vol. in gases. Coulometric titrations are with electroytically generated bromine. G. SKIRROW

2901. **Development of automatic titrators.** J. R. Maher (*Chem. Age*, 1954, **70**, 1441–1446, 1448).—Fields of development discussed in detail are potentiometric determination of end-points, photometric determination, electrolytic generation of titrants in the standardisation of solutions, and thermometric titration; end-point determination by chemiluminescent indicators and high-frequency titrimetry are dealt with briefly. G. HELMS

2902. **Improved method and apparatus for determination of moisture content of fibrous or other materials.** J. L. Shaw (Brit. Pat. 709,177; Date Appl. 4.11.52).—The moisture content of closely packed materials, such as cheeses, cops or other packages, is determined by measuring the capacitance between the needles of a needle electrode system inserted into the material. Results are accurate even when the moisture is unevenly distributed throughout the samples. D. R. GLASSON

2903. **A simple electromagnetic gas-valve.** J. T. Stock and M. A. Fill (*Lab. Practice*, 1954, **3** [8], 335–336).—The construction and operation of a gas-valve for temp. control of a heating-block or micro-oven as used for sealed- or capillary-tube organic preparations and reactions is described and illustrated. The solenoid-actuated valve can be combined with a mercury, mercury - toluene, or similar regulator, so that the contacts close when the required temp. is reached. A buffer-action micro-burner is included in the circuit to prevent extinction of the flame owing to the rapid opening and closing of the valve, which is operated from a 4 V supply. W. J. BAKER

2904. **Improvements in or relating to identification or analysis of substances.** Lantson Ltd. (Brit. Pat. 710,373; Date Appl. 24.9.51).—The substances are subjected to electromagnetic waves generated within the radio-frequency band. For single substances, the frequency of max. energy absorption directly depends on the quantity of substance present. In mixtures of substances, *e.g.*, where impurities are present, changes (*a*) in resonance frequency or (*b*) in energy absorbed at resonance with no change in resonance frequency may occur. Change (*a*) alone results on addition of organic liquids to water or to each other; (*b*) results when inorganic salts are added to water. Examples studied include H_2SO_4, HNO_3, HCl, methanol, ethanol, acetic acid, NaCl and mineral oil. D. R. GLASSON

2905. **Mass spectrographic analysis of solids.** N. B. Hannay and A. J. Ahearn (*Anal. Chem.*, 1954, **26** [6], 1056–1058).—The preparation and

mounting of solid specimens for a double-focusing mass spectrometer are described. Cylindrical rods or compressed powder contained in small tubes (0·03 to 0·04 in. diameter, 0·25 to 0·50 in. long) are etched to remove surface impurities and are exposed end-on to the electron source. Several samples are examined and the results are recorded photographically. The spectra are used for qualitative and semi-quantitative analyses, and when mixtures are examined, a line originating from the principle component is used as an internal standard for determination of minor constituents. Examples include: Ge containing 6 p.p.m. of Sb, Sb containing 100 p.p.m. of As, Cu bearing a monolayer of Au or In, and B in Si. D. A. PANTONY

2906. **A mass spectrograph for the analysis of solids.** N. B. Hannay (*Rev. Sci. Instrum.*, 1954, **25** [7], 644–648).—A Mattauch-type mass spectrograph designed for either photographic or electrical ion detection is described. The instrument has been applied to the analysis of impurities in solids and bulk concentrations $< 0{\cdot}1$ p.p.m.; surface contaminants of $< 0{\cdot}1$ monolayer can be detected. G. SKIRROW

2907. **An improved mass spectrometer ion source.** O. A. Schaeffer (*Rev. Sci. Instrum.*, 1954, **25** [7], 660–662).—The ion source for a 60° mass spectrometer has been improved by almost completely enclosing the ionisation region to reduce the contribution of ions from thermal decomposition products on the filament, and by a built-in stronger magnetic field. G. SKIRROW

2908. **Process monitor mass spectrometer.** J. K. Walker, A. P. Gifford and R. H. Nelson (*Ind. Eng. Chem.*, 1954, **46** [7], 1400–1403).—A portable 180° mass spectrometer is described; it is designed for the continuous measurement of one or more components in a process gas stream. The concentrations are presented directly as mole percentages. Provision is made for automatic periodic standardisation against normal stream composition or stored samples of the pure gases. G. SKIRROW

2909. **Ion-resonance mass spectrometer. Industrial applications.** W. A. Morgan, G. Jernakoff and K. P. Lanneau (*Ind. Eng. Chem.*, 1954, **46** [7], 1404–1409).—An ion-resonance spectrometer is described; it is suitable for continuous process control and for use as a routine gas analyser, leak detector and trace constituent analyser. High sensitivity and good resolution are given over the mass range 2 to 100. G. SKIRROW

ERRATA.—April (1954) issue, abstract 758, last line.
The first word should be "involving".

April (1954) issue, abstract 792, third line from bottom.
For chichonine *read* cinchonine.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
atomic	at.
boiling-point	b.p.
British thermal unit	B.Th.U.
calculated	(calc.)
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
critical	crit.
crystalline, crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment, -al	expt.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
indefinite	indef.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim -um, -a	max.
melting-point	m.p.
microgram	μg
micro-litre	μl
micron	μ
milliampere	mA
milligram	mg
millilitre	ml
millimetre	mm
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
organic	org.
ounce	oz
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative humidity	R.H.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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ANALYTICAL ABSTRACTS

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